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JOURNAL OF

METALS

Vol. 13, No. 8
August, 1961

COVER: by David Cain
For a change—the cover has absolutely nothing to do with this issue or with metallurgy; it represents our thoughts (and maybe yours too) which have been drifting toward this cool, mountain lake with each passing day of summer. Inside the cover there's information on scandanium, vanadium, and beryllium plus other articles.

contents 517
engineering consultants 518
personnel 518
books 519
personals 524
letters to editor 526
editorial 527
reporter 531
education 534
news of your profession 575
around the sections 576
advertisers index 580
coming events 580

RECENTLY ACCEPTED TRANSACTIONS PAPERS 523

Beryllium Processing by Powder Metallurgy 537
by K. G. Wikle and V. C. Potter

Gaseous Reduction of Oxygen-Containing Copper 545
by Leonard Klein

Decarburization by Open-Coil Annealing 548
by D. J. Blickwede

Scandium Recovery From Uranium Solutions 555
by L. D. Lash and J. R. Ross

Part V. History of the Coking Industry in the United States 559
by C. S. Finney and John Mitchell

Extractive Metallurgy of Vanadium 562
by Blair Burwell

Latin-American Iron and Steel Congress 567
by Robert W. Shearman

Agglomeration—First International Symposium 571

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ADVERTISING SALES MANAGER: Lee Kent, 122 East 42nd St., New York 17, N. Y.
WESTERN ADVERTISING REPRESENTATIVE: Dillenbeck-Galavan Inc., 266 S. Alexandria Ave., Los Angeles 4, Calif.

Address insertion orders and copy to: Advertising Department, JOURNAL OF METALS, 29 West 39th Street, New York 18, N. Y. Send plates to: JOURNAL OF METALS, C/O Lew A. Cummings Co., Inc., 215 Canal St., Manchester, N. H. Published the first day of each month by the American Institute of Mining, Metallurgical and Petroleum Engineers, Inc., 29 West 39th Street, New York 18, N. Y. Telephone: PEnnsylvania 6-9220. Subscription $10 per year for non-AIME members in North, South & Central America, $12 elsewhere; $6 for AIME members, or $4 additional for members, only in combination with a subscription to "Mining Engineering" or "Journal of Petroleum Technology." Single copies, $1.50. The AIME is not responsible for any statement made or opinions expressed in its publications . . . Copyright 1961 by the American Institute of Mining, Metallurgical, and Petroleum Engineers, Inc. . . . Registered address, AIME New York . . . Indexed in Engineering Index, Industrial Arts Index, and Chemical Abstracts . . . Second class postage paid at New York, N. Y., and at Manchester, N. H. Numbers of copies in this issue, 12,100.

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If you are interested in any of these listings, and are not registered, you may apply by letter or resume and mail to the office nearest your place of residence, with the understanding that should you secure a position as a result of these listings you will pay the regular placement fee. Upon receipt of your application a copy of our placement fee agreement, which you agree to sign and return immediately, will be mailed to you by our office. In sending applications be sure to list the key and job number.

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Concentrator Superintendent, Assistant-Copper, age 36, MetE, AIME. Recently superintendent of 15,000 tpd copper mill, moly plant, 1200 tpd CCD slime leaching plant. Formerly foreman of 200 tpd contact acid plant, foreman of tank house for electrodeposition and electro-refining of copper. Prefer Western United States. Home: Arizona. $900. Se-192.

Metallurgist, Assistant Superintendent-Ore Processing, age 32, MetE, AIME. Over four years testing and control of various phases of process in zinc smelting (horizontal sintering machines, retort smelting, magnetic separation) and processing copper ore (crushing, grinding, flotation, reagent preparation and control) and processing tungsten ore (crushing, grinding, tabline). Prefer South America. Home: Colorado. $7500. Se-140.

Consultant, MetE, AIME, age 70. Twenty years experience in private commercial production of non ferrous hi-purity ingot metals in United States and foreign countries. Twenty-five years research and development extractive process metallurgy. Prefer Southwest United States. Home: Nevada. $400. Se-1241.

Metallurgist, age 38, MetE, AIME. Two years experience in melting procedures, laboratory operations, sand control, advisor on welding and other technical problems on iron and steel. Two years experience in metallurgy and brazing on research program for brazed titanium honeycomb panels, consultant on ferrous castings regarding their specification, procurement, heat treatment, usage. Two years in charge of experimental stress analysis lab, supervised all metallurgical aspects of making, heat treating, matching and testing of experimental castings. Prefer Western United States, Foreign. Home: Illinois. $13,000. Se-1483.

Research Development Engineer, age 24, MetE, AIME. Recent graduate with some experience in drafting and designing in silver, lead, copper plants. Prefer Western United States. Home: Canada. $7000. Se-1379.

Metallurgist, age 27, MetE, AIME. Eighteen months metallurgical chemist in zinc plant including production, quality control, laboratory work. Prefer Western United States, Home: Idaho. $7000. Se-1651.

——POSITIONS OPEN——

Engineers. (c) Metallurgist, recent graduate, particularly familiar with metallography examination, cross section alloying, etc. for product development. (d) Metallurgical or Mechanical Engineer, graduate, familiar with basic metallurgy to train to take charge of one section of rolling and cladding. New York Metropolitan area. W466.

Staff Metallurgical Engineer with at least 5 years experience covering design and layout of copper ore handling and metallurgical processing equipment. Salary open. Near East. F481.

Consultant for six month to one year assignment in Germany. Must have specialized in the field of sintering. F465.

Sr. Research and Development Metallurgists. (a) Ten years experience on welding and ferrous materials on failure analysis, analyze and evaluate welds and welding processes, heat treating and annealing. (b) Ten years experience on refractory materials particularly tungsten, to guide a plasma gun program aimed at producing rocket nozzle components. Develop technique required for manufacturing rocket nozzle components from refractory materials. $12-$15,000. Northern N. J. W441.

Sales Engineer, background in metals and metallurgy, with extensive contacts with steel companies and ferrous alloy producers. International trade background with knowledge of ferrous ores. Some travel. To $10,000. Headquarters, New York City. W440.

Engineers. (a) Manager of Metallurgical Development-Marketing; B.S. or M.S. in ferrous metallurgy with 5 to 10 years experience, including actual steel production experience with open hearth or electric furnace operation; should have had successful selling experience with ferro alloys, and should be familiar with these industries. Through knowledge of the field and broad contacts vitally important. Will be concerned with production applications in the steel industry with concentration at the operating level. Will demonstrate products, new and old, on the open hearth or electric furnace floor. To $12,000. (b) Sales Engineer, degree in metallurgy, with 2 to 3 years experience in the electric furnace or open hearth steel industry. Some selling experience beneficial. Semi-trainee job. $7000-$9000 year to start. Apply by letter including present and desired salary. Pennsylvania. W437.

Staff Metallurgist, 35-50, with at least B.S. in metallurgy, chemial engineering, chemistry or mining, with experience in chemical laboratory, inorganic, desirable; metallurgical research on zinc, lead, copper, tin or chemical desirable; operating supervision, zinc, lead, copper, tin or chemical desirable; design and construction of metals or materials handling equipment. Will observe and criticize operating procedures, recommend improvements, keep abreast of new developments in related industry; develop and propose operating procedures for new metallurgical projects, etc. $10,000 year plus liberal benefits. Midwest. W435.

Sales-Castings, $500 up, in San Francisco Area, ME, MetE, young. Engineering background; some sales experience and aptitude. Sell precision castings used on jet engines, conveyors to industry in a developed territory (used by manufacturers of cans to missiles). Should have management capabilities for possible later company representation. For a manufacturer. SJ-6223.

Research Metallurgist, B.S. in metallurgy, preferably Ph.D. in metallurgy, age 25-45. Will plan, perform, analyze and report research on development of new or improved aluminum alloys or fabricating processes for aluminum. $9300-$12,800. Employer will pay placement fee. Northwest U.S. C8698.

Metallurgist R&D, graduate plus post graduate work. 25-35. Must have good theoretical background to approach problems; some experience in either X-ray diffraction work or precision casting desired for an automotive and truck parts manufacturer. Research and development leading to development of new metallurgical processes and products. Salary open depending on experience. Northwest suburb of Detroit. C8705.

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books ANNOUNCEMENTS AND REVIEWS

Books that are marked (•) may be ordered through AIME. Address Irene K. Sharp, AIME Book Dept., 29 W. 39 St., New York 18, N. Y. A discount is given whenever it is possible.

Fermi Surface

The Fermi Surface, edited by W. A. Harrison and M. B. Webb, John Wiley & Sons, Inc., New York, 356 pp., $10.00, 1960. *Reviewed by C. Ernest Birchenall, University of Delaware.* •

The papers and discussions recorded in this book constitute the proceedings of a conference on Fermi surfaces of metals. The list of participants published ahead of the Table of Contents guarantees that the contents represent the carefully considered opinions of those in the forefront of the theoretical and experimental study of the energy states of electrons in metals. The abundance of new material reported at the conference is an index of the intense activity in this field. The exploration of new experimental approaches, their utility and limitations, is described in these pages. The crisis in theory which leads some to ask seriously "is there a Fermi surface?" and others to proceed "from the hypothesis that a Fermi surface exists and may be measured" is generating a thorough re-evaluation of the foundations of the subject, of its assumptions and procedures which have been dignified by constant repetition.

The state of affairs so clearly shown by these papers must certainly excite the solid state physicist to a detailed reading and careful evaluation of every word. The average metallurgist is not prepared to follow the arguments in detail, but the metallurgist can ignore this revolution only at considerable peril. For instance, the physical metallurgist can and should take refuge in the recollection that the Hume-Rothery electron-atom ratio rules were derived from the systematic examination of a large number of experimental cases. Later a theory based on nearly spherical Fermi surfaces in the monovalent noble metals was invented to explain the observations. This picture was considerably modified several years ago by the theoretical work of Friedel. Now that the Fermi surfaces have been found to be much more complicated than they had been assumed to be, it seems possible that the *rationale* of Hume-Rothery's rules will undergo major surgery.

The papers are grouped in the following categories: theory, the de Haas-van Alphen effect, galvanomagnetic effects, cyclotron resonance, anomalous skin effect, magnetoacoustic effect, and transport properties and studies of alloys.

Many readers may find it helpful, as the reviewer did, to read the summary articles at the end of the book before reading the papers.

This book is recommended reading for those metallurgists with a strong background and interest in solid state physics. Others can more profitably wait for further development of this active field and for the simplified and more integrated accounts which will surely begin to appear soon.

* * *

Management, Organization, and Methods in the American Iron and Steel Industry, published by the European Coal and Steel Community, 360 pp., $3.00, 1961.

This is the final report of the fact-finding team sent to the United States by the High Authority of the European Coal and Steel Community (ECSC) in 1957 to study management organization and methods as productivity factors in the American iron and steel industry. The team consisted of managing directors and other qualified men from the six member-countries of ECSC.

The English translation of the report is divided into two parts. In Part One, the authors seek to pinpoint the specifically American factors which they consider likely to have affected the management arrangements ultimately adopted.

Part Two examines management in the American iron and steel industry firstly as a job (concepts, methods, and setups), and secondly as a group of persons (staff rules and regulations, functions, personnel strengths).

Proceedings, Third Symposium on Electron Beam Technology, edited by Robert Bakish, published by Alloyd Electronics Corp., 379 pp., $15.00 1961. •

This volume contains the proceedings of the conference held March 23-24, 1961 in Boston. The book is divided into four sections.

The first section, *Physics of Electron Beams,* covers gun design, gun material limitations, and other fundamental parameters in theoretic electron beam systems.

Section two, *Electron Beam Welding,* presents the most recent data on electron beam welding as obtained from studies with both high and low voltage systems.

Less Known Electron Beam Applications, which is section three, covers the topics of machining, electron beam recording, and the role of electron beams as media for generating of radiation changes.

The last section, *Electron Beams in Microminiaturization,* discusses progress in the technology, such as microelectronics and related topics.

Copies of the proceedings are available from Alloyd Electronics Corp., 35 Cambridge Parkway, Cambridge 42, Mass.

High-Strength Steels for the Missile Industry, edited by H. T. Sumsion, published by American Society for Metals, 276 pp., $12.00, 1961. •

This book is comprised of papers presented at a symposium held during the Golden Gate Metals Conference in San Francisco February 4-6, 1960, and sponsored by ASM.

There are 10 papers by 14 authors concerning problems in development and application of ultra-high strength metals for the U.S. missile program.

Two publications of interest to metallurgists have recently been announced by Technical Translations, P.O. Box 157, Altadena, Calif.

Blast Furnace Control by Computers, by V. Loskutov, Promysh. Ekonom. Gazeta, v. 5, 3 pp., 1960, $2.90, Order No. 5032.

This is a brief report on Soviet developments in the field of blast furnace control by computers. The general principle involves: advantages of breaking down the blast furnace process into zones, using a series of computers; automatic continuous computation of the direct-reduction coefficient; coordinating control computer as a final step to integrate the entire system of zonal control; and computing devices.

Blast Furnace Operation on Natural Gas, by M. A. Shapovalov, Metallurg, No. 8, 1960, 1800 words, 1960, $2.90, Order No. 5124.

This report discusses the importance of proper use of natural gas in the blast furnace for economical operation. It also lists the advantages of increased hydrogen content in hearth gases even at the expense of cutting the CO content. Also discussed are the merits of the use of coke gas in the blast furnace over use in the open hearth furnace, CO_2 distribution over cross section below stockline in various blast furnaces, and the effect of CO in top gas on carbon utilization coefficient with various limestone additions.

(Continued on page 522)

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The Proceedings of a Technical Symposium sponsored by the Nuclear Metallurgy Committee of the Institute of Metals Division, and the Titanium, Uranium, and Uncommon Metals Committee of the Extractive Metallurgy Division, The Metallurgical Society of AIME. San Francisco, February 16-17, 1959.

Edited by W. D. WILKINSON, Argonne National Laboratory

The addition to these Proceedings of an introductory chapter on the metallurgy of plutonium and of an extensive annotated bibliography has made this volume of even greater value to metallurgists working in this particularly important field. Mr. Wilkinson both edited the Proceedings and provided the new material.

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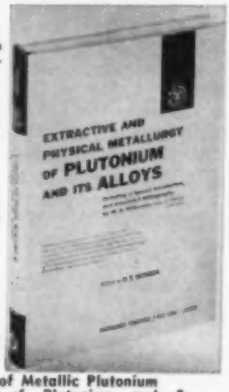

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edited by **GEORGE R. ST. PIERRE**

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Extractive Metallurgy of Copper, Nickel, and Cobalt

Based on an International Symposium sponsored by the Extractive Metallurgy Division of The Metallurgy Society of AIME; New York, February 15-18, 1960

edited by **PAUL QUENEAU** including an Annotated Bibliography by **Ken G. Robb**

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Volume 9 of Metallurgical Society Conferences

Sponsored by the Physical Metallurgy Committee, Institute of Metals Division, The Metallurgical Society of AIME, Estes Park, Colorado, July 11-12, 1960

edited by P. G. SHEWMON and V. F. ZACKAY

CONTENTS

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May, 492 pages **Members: $14.40, Nonmembers: $18.00**

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Books

(Continued from page 519)

Proceedings of the Sixth Conference on Magnetism and Magnetic Materials, McGraw-Hill Book Co., Inc., 395 pp., $10.00, 1961. •

This volume contains 149 papers presented at the Magnetism Conference held in November 1960. The conference was jointly sponsored by the American Institute of Electrical Engineers and the American Institute of Physics.

Groups of papers dealing with pure physics and applications provide a review of the current state of both theory and practice, and cover the topics of ordered spin systems; computer devices; spin configurations; metallic films; nuclear hyperfine fields; ferromagnetic resonance; high coercive materials; exchange interactions and fine particles; anisotropy; domain walls and domain wall motion; microwave devices; metals and alloys; soft magnetic materials; and oxides.

The Engineering Industries In Europe, published by the Organization for European Economic Cooperation (OEEC), 287 pp., $1.75, 1961.

This is the fourth study by the OEEC's Machinery Committee in the series, *Trends in Economic Sectors.* This study covers those branches of the metal processing industries which produce non-electrical machinery; electrical machinery, apparatus and appliances; transport equipment; manufactures of metals, precision instruments, watches and clocks. This report covers the period from the beginning of 1957 to the end of 1959, and a qualitative assessment of the situation is made, as far as possible, up to the closing months of 1960.

Part I of the report is a summary review of trends. Part II analyzes the principal factors of supply, while Part III concerns the principal factors of demand. Part IV concerns foreign trade.

Copies of the report are available from the OEEC Publications Office, 1346 Connecticut Ave., N.W., Washington 6, D. C.

World's Nonferrous Smelters & Refineries 1960 (Including a Classified Guide to the Metals and Alloys Produced), Sixth edition, edited by H. G. Cordero, Quin Press Ltd. (London), 451 pp., $11.20, 1960. •

This publication lists the nonferrous smelters and refineries by country and continent. Information about each company includes the year it was established, location of the head office, directors, capital, properties and plants, products, process and raw materials used, brands or descriptions, sales offices, and associated and subsidiary companies.

A new feature of the book is a directory listing the producers of nonferrous metals and alloys.

Statistics of World Trade in Steel 1913-1959, prepared by the Secretariat of the Economic Commission for Europe (ECE), published by the United Nations, 477 pp., $6.00, 1961. •

This report consists of tables showing exports of semifinished and finished steel products by regions and countries of destination.

The data on exports contained in this publication have been compiled by the Secretariat of the UNECE from information and statistics collected for a recent report, *Long Term*

(Continued on page 578)

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Effect of Temperature on Yielding in Single Crystals of the Hexagonal Ag-Al Intermetallic Phase, by J. D. Mote, K. Tanaka, and J. E. Dorn (IMD)

The Fracture Behavior of Silver Chloride-Alumina Composites, by T. L. Johnston, R. J. Stokes, and C. M. Li (IMD)

Observations of the Structure of Aluminum Specimens Grown from the Malt, by P. E. Doherty and R. S. Davis (IMD)

Zinc-Rich Corner of the Zn-Fe-Al System, by E. H. Rennback (IMD)

Structural Relationships between Precipitate and Matrix in Cobalt-Rich Cobalt-Titanium Alloys, by R. W. Fountain, G. M. Faulring, and W. D. Forgeng (IMD)

Effects of Grain Boundary Structure on Precipitate Morphology in an Fe—1.55% Si Alloy, by S. Toney and M.I. Aaronson, with an appendix by N. A. Gjostein (IMD)

Effect of Surface Removal on the Plastic Behavior of Aluminum Single Crystals, by I. R. Kramer and L. J. Demer (IMD)

The Cadmium-Uranium Phase Diagram, by Allan E. Martin, Irving Johnson, and Harold M. Feder (IMD)

The Columbium-Oxygen Equilibrium in Liquid Iron, by Michel Elle and John Chipman (ISD)

Magnetism in a High Carbon Stainless Steel, by S. M. Purdy (IMD)

The Oriented Growth Mechanism of the Formation of Recrystallisation Textures in Aluminum, by M. N. Parthasarathi and Paul A. Beck (IMD)

Activities of Oxides in CaO-FeO-Fe_2O_3, by E. T. Turkdogan (ISD)

Derivation of Phase Diagram for the Silicon-Oxygen-Carbon System, by W. A. Krivsky and R. Schuhmann, Jr. (EMD)

Measurement of the Surface Self-Diffusion Coefficient of Copper by the Thermal Grooving Technique, by N. A. Gjostein (IMD)

Silver Diffusion in the Intermetallic Compound AgMg, by W. C. Hagel and J. H. Westbrook (IMD)

The Zirconium-Rich Corners of the Ternary Systems Zr-Co-O and Zr-Ni-O, by M. V. Nevitt and J. W. Downey (IMD)

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Acceleration of Deformation by Concurrent Phase Change, by A. G. Guy and J. E. Pavlick (IMD)

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The Influence of the Temperature of Deformation on Strain Aging of Alpha-Iron, E. Lautenschlager and J. O. Brittain (IMD)

Sigma Phases with Aluminum, by K. P. Gupta (IMD)

Observations on Twinning in Zone-Refined Tungsten, by H. B. Probst (IMD)

Determination of Interstitial Solid-Solubility Limit in Tantalum and Identification of the Precipitate Phases, by Dale A. Vaughan, Oliver M. Stewart, and Charles M. Schwartz (IMD)

Thermodynamic Considerations in the Chlorination of Different Oxides Constituting Columbite (Niobite) and Tantalite, by G. V. Jere, C. C. Patel, and V. Krishnan (EMD)

Strain Aging Effects in Arc-Cast Molybdenum, by G. W. Brock (IMD)

Some Recovery Characteristics of Zone-Melted Iron, by J. T. Michalak and H. W. Paxton (IMD)

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Spectrochemical Slag Analysis with the Tape Technique, by A. Danielsson, I. Nilsson, and G. Sundkvist (EMD-ISD)

Zone Purification of Beryllium, by W. R. Mitchell, J. A. Mullendore, and S. R. Maloof (EMD)

Kinetics of the Platinum-Catalyzed Hydrogen Reduction of Aqueous Cobalt Sulfate-Ammonium Acetate Solutions, by R. Ted Wimber and Milton E. Wadsworth (EMD)

TECHNICAL WRITER

* * *

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PHYSICAL METALLURGIST CHEMIST

Important new, long-range projects at the IBM Thomas J. Watson Research Center at Yorktown, N. Y., have created an outstanding opportunity for a person with an advanced degree or equivalent experience in physical metallurgy or chemistry. The person who can fill this position must be able to take charge of a semi-conductor crystal-growing activity and carry out program of research on related problems. Experience should include preparation of solid state materials and a background in semiconductor physics.

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personals

Paul W. Beamer has been appointed manager of sales for the eastern div., Wyman-Gordon Co. He will also continue as director of product and sales development.

William H. Bickley, technical information specialist, has joined the staff of Clyde Williams and Co., in Columbus, Ohio. He was formerly with Battelle Memorial Institute.

Ardrey M. Bounds, chief metallurgist with Superior Tube Co., and **Bruce W. Gonser,** technical director, Battelle Memorial Institute, have been elected for three-year terms on the board of directors of the American Society for Testing Materials.

Claude L. Clark, staff metallurgical engineer, tubing applications, for Timken Roller Bearing Co., has been chosen to receive the 1961 Albert Sauveur Achievement Award of the American Society for Metals. The award was established by the society in 1934 to recognize pioneering metallurgical achievements which lead to advances in metallurgical engineering.

Lawrence N. Collins has been appointed assistant sales manager of Arthur G. McKee & Co.'s Metals div. He joined the company in 1956 as manager of the consulting department.

R. S. Cremisio is now a Fellow-Solidification Metallurgy, at Mellon Institute. He was formerly a staff metallurgist with Crucible Steel Co. of America. He is also completing the requirements for a Ph.D. at the University of Pittsburgh.

Andrew Fletcher, chairman of St. Joseph Lead Co, has been reappointed for a four year term, expiring in October 1965, as one of AIME's representatives on the Board of the United Engineering Trustees.

John W. Goth has been appointed manager of foundry sales for Climax Molybdenum Co. He was formerly head of the company's Chicago sales office.

Robert R. Hendren has become president of Graftex Products, Inc., Riverton, N. J. He was previously general sales manager of Hoeganaes Sponge Iron Corp.

Franz R. Hensel has joined Clyde Williams and Co., as executive vice president. He will also be president of Clyde Williams Corp., a subsidiary. He was formerly vice president of engineering of P. R. Mallory and Co., Inc.

(Continued on page 526)

CANADA WKE is responsible for marked progress in aggregate preparation. Long experience in mining, handling, concentration and storage of ores and minerals produced this 900 TPH sand and gravel plant.

MEXICO This fluid cat cracker is the latest of many major units engineered by McKee in the extensive expansion of Mexico's petroleum industry. McKee's long experience in Mexico was invaluable here. Our experience in your country can prove equally valuable on *your next project*.

ENGLAND Clean-cut design of this blast furnace plant shows the quality of engineering you can expect from McKee-WKE. As early as 1939 McKee set a pattern for the industry with a British ore beneficiation plant of revolutionary design. We can apply thorough, experience-backed knowledge like this to your project—*anywhere*.

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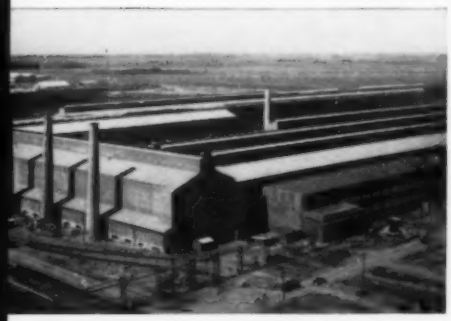
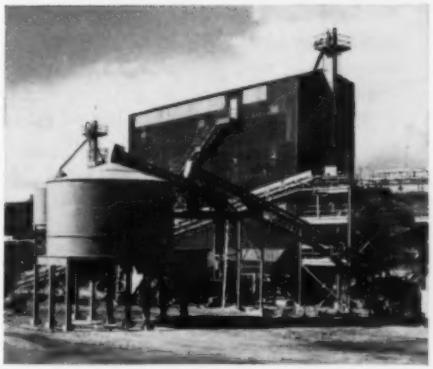
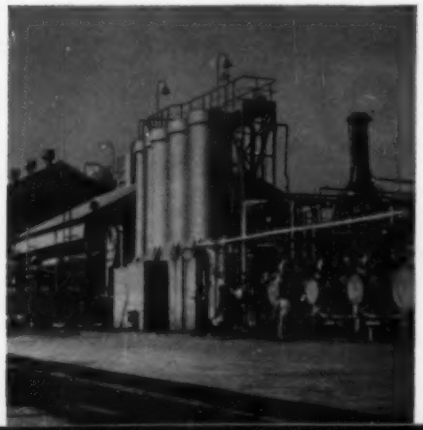




ARGENTINA Decades of steel making knowledge by McKee went into the design and construction of this 700-acre steel plant. New work in progress by McKee will give it largest capacity in Latin-America.

JAPAN Distance was no problem here. McKee engineers added this benzol-acetone solvent dewaxing unit to vacuum distillation and fluid catalytic cracking units designed and built by McKee in Yokohama.



UNITED STATES Thorough knowledge of production methods, from raw material to finished product, were applied in this WKE-built plant which produces milk of lime for use in concentration of copper ore. WKE also designed and built a concentration plant and a copper smelter in this area. Knowledge like this can save you time and money.

BRAZIL Steel men everywhere rely on McKee technical know-how. South America's first large integrated steel plant was designed, built and later expanded by McKee. Operated by McKee-trained local personnel, the two-mile-long plant is an outstanding success. The McKee-designed sintering plant at left was completed recently.

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SUBJECT:

carbon

Process metallurgy of carbon reduction

We will explore here only the process metallurgy associated with carbon reduction. With oxygen practice becoming the rule in open hearth shops, carbon can be reduced to very low levels. But, as many people using electrolytic manganese today can testify, carbon aims are not the whole story. To see why, consult first the iron-carbon diagram, and second, the chart showing increased absorption of oxygen in liquid steel with increasing temperatures.

In order to achieve extra-low carbon levels, it is necessary to raise bath temperature, which in turn means that more oxygen will be absorbed by the liquid steel. At tap, temperature and oxygen levels are not reduced, although carbon is raised by the ladle addition.

Extra Oxygen Content Requires Stepped-up Use of Deoxidizers

The effects of high oxygen residuals upon surface are still being explored. One effect of extra oxygen is to require increased amounts of metallic deoxidizer, with a consequent increase in non-metallic inclusions.

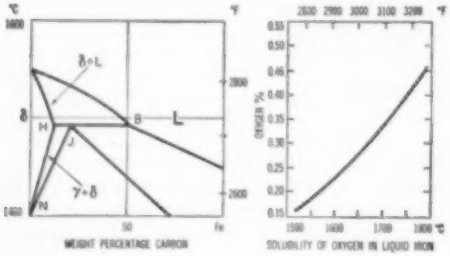
Surface Quality of Steel Suffers with Higher Temperatures

Effects of high temperature upon surface are well-known. The old rule-of-thumb is "lower temperatures—better surface" . . . for steel made by any method. Thickness of the first-formed "skin" of an ingot is relative to heat-absorbing ability of the mold wall, and the temperature of the steel. In identical molds, a high temperature heat will not form as thick a skin as a relatively low temperature heat. An ingot with a thick surface skin is better able to withstand stresses of cooling, and will not tear as easily as one with a thin skin.

Foote Electromanganese Pays Own Way in Better Quality

Foote electrolytic manganese pays for itself in many varied ways. Some advantages are not always obvious, of course. However, with ever-increasing pressures to produce cleaner, better-surface steels, even a small advantage in quality and performance can pay off in lowered plant costs, lowered claim costs and greater customer satisfaction.

For more data on Foote Electromanganese®, write for Bulletin 201, Foote Mineral Co., 481 Eighteen West Chelten Ave., Phila. 44, Pa.



FOOTE
MINERAL COMPANY

letters to the editor

First D-H Unit

Your article, *America's First D-H Unit*, on page 431 of your June issue begins with the sentence: "The world's largest D-H degassing unit—the only one of its kind in the US—was recently demonstrated by the Crucible Steel Company. . ."

The Crucible unit is not the only one of its kind in the United States. We also have a D-H unit, of smaller capacity than the Crucible unit, which has been in successful operation for several months.

We would very much appreciate your printing a correction in your next issue.

Ralph Kaplan, Manager
Market Research
National Forge Co.
Irvine, Pa.

Personals

(*Continued from page 524*)

Michael A. Kuryla has been appointed manager of the Lima (Peru) div. of Cerro de Pasco Corp. He was formerly deputy manager of the division.

Daniel A. Porco and **Eugene A. March** have been elected vice presidents of Crucible Steel Co. of America. Mr. Porco was formerly assistant to the president, and Mr. March was director of technology.

John R. Purdy has been appointed manager of the Colorado Fuel and Iron Corp.'s Pueblo coke plant. He was formerly assistant superintendent.

Lazarus C. Weiner is now vice president of E. J. Doucett Associates, Summit, N. J. The company is engaged in research and development in the fields of electronics, physics, materials, and chemistry.

Franklin H. Wilson has been appointed head of the physical metallurgy research div. of Anaconda Co., and **David H. Thompson** has been appointed head of the chemical metallurgy research div.

Harry Czyzewski, president of Metallurgical Engineers, Inc., has been appointed a member of the Oregon State Board of Engineering Examiners. He is the first metallurgical engineer appointed to the board, although several mining engineers have served in the past.

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editorial

A Solution to Engineering Education

[Last month, the training of engineers was discussed in this column; this month some solutions are offered to the perplexing problems posed, as we conclude our excerpts from an address by Philip Sporn, president of American Electric Power Co. Ed.]

"There is a great need, I think, for our colleges to comprehend the idea that the 1960-1965 student body will not make the world of 1965, but will have the responsibility for creating the world of 1985. It is for this that they must be trained. . . "

"There is something too, we could do about the student body entering our engineering schools. . . We do not get a large enough share of our talented youth. I believe that we must find some way to attract more of our young people to engineering schools, and we need to do a great deal of what I can best describe as beneficiation before we bring this talent into our engineering schools. Essentially, I believe, this requires a re-orientation of both the character and the tempo of training in our high schools and preparatory schools. . . "

"The curricula that we establish for the people we bring into our schools . . . need to be deepened and broadened. . . The student needs to be given at least a start toward understanding the world he is going to work in, the world he will try to improve, and the world he may have the job of running. He needs to have solid background to have full awareness not only of all the aspects of the big technological problems he is working on and trying to solve, but of how they tie in with attempts by others to produce a better social and material environment. In short, since he is going to be called upon to reach decisions that may have far-reaching effects on society, not only must he be capable of crossing traditional dividing lines in engineering, but he must develop a deep understanding of the social-economic forces at work in our own country and in the world.

"We need to do some clear thinking about specialization. Specialists are needed, but the specialized skills can be developed much more easily at a later period than can the breadth and depth. These latter must come earlier. No engineer, for example, should be permitted to go out into the world without fairly solid grounding in geography and oceanography; or in meteorology, geology, and hydrology. And surely he cannot do without a knowledge of engineering materials, structural chemistry, or energy sources and energy conversion systems. We need to look at the problem of engineering training in terms of a time period that extends well beyond the demands of the moment for certain highly specialized skills."

"I believe the students in our engineering schools, if they are going to be engineers and not merely highly successful, even brilliant, technicians, need to have a great deal of contact with engineering. They need to have much greater contact with industry. . . We need more . . . (programs that) . . . bring students into contact with engineers at an early phase of their careers. . . It gives them a much more solidly based appreciation of engineering and of a career in engineering.

"I think we need to do something about faculties. I believe students of engineering need to be inspired to climb to the heights of engineering: if they are going to be great engineers they ought to be exposed to at least a few great engineers. . . I believe this can be rectified by bringing some practicing engineers into our engineering faculties. There is a good technique for this—the adjunct professorship—which should be developed."

"Well, this is quite a list of things we need to do. They are, I believe, *do-able*. And if we succeed . . . then we will have trained and produced the engineers we must have in our society if we are to solve the terrifically difficult national and world problems confronting us today and which will confront us to an even greater degree tomorrow."

ANGLES BETWEEN PLANES IN CUBIC CRYSTALS

By R. J. Peavler and J. L. Lenusky

IMD Special Report Series Number Eight

This small but valuable 28-page booklet contains tables of interplanar angles in the cubic system up to {554}. Calculated on a digital computer by members of the Crystallographic Group at Westinghouse, the values were checked carefully to prevent error in transcription. Typographical errors were eliminated by reproducing the original tables. Invaluable for orientation work in many phases of physical and X-ray metallurgy. Author R. J. Peavler is a crystallographer in the Crystallographic Group of the Materials Engineering Departments, Westinghouse Electric Corp. J. N. Lenusky is a former laboratory assistant in the Crystallographic Group.

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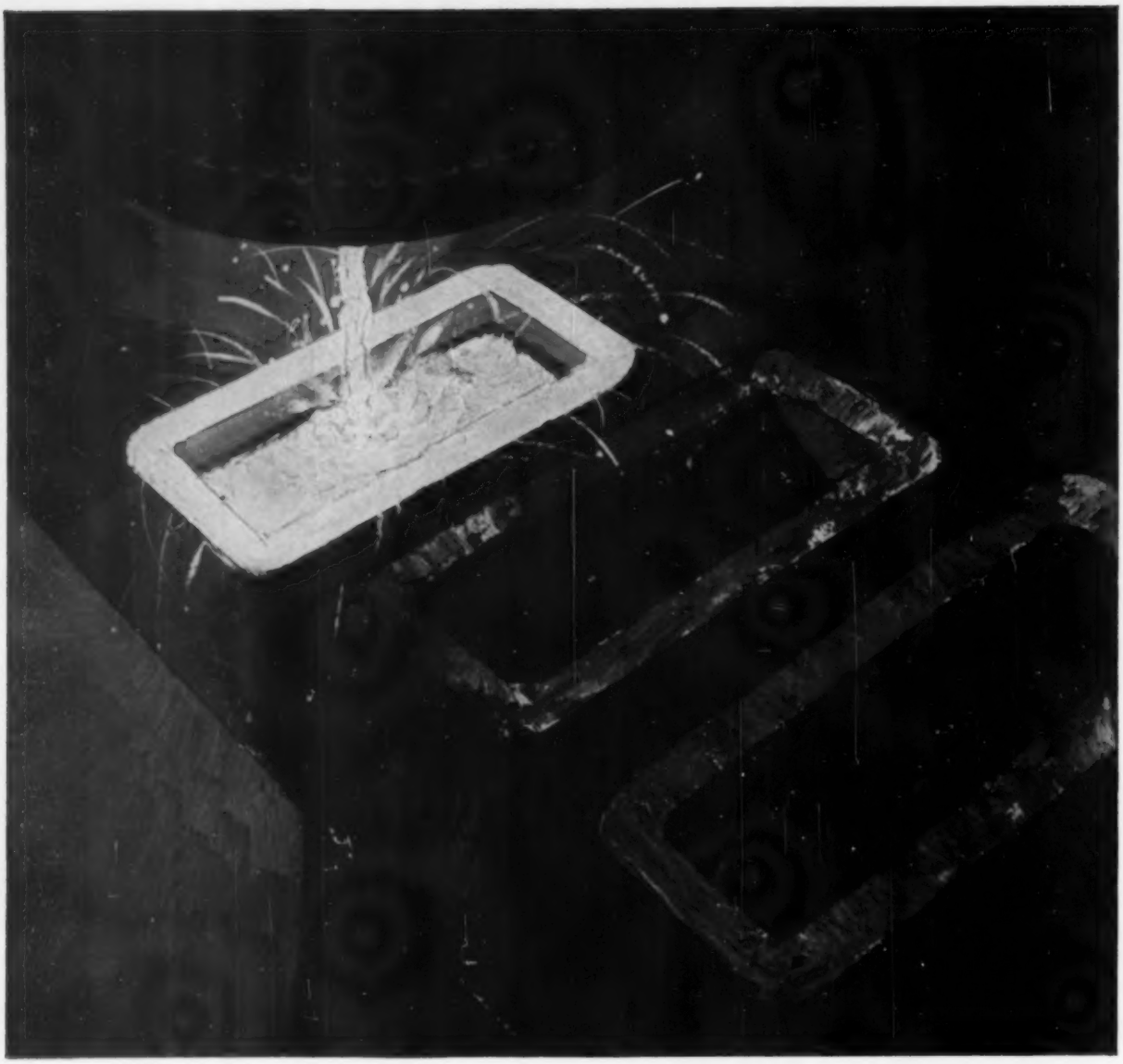

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CF&I oxygen steel

The Colorado Fuel and Iron Corp. recently tapped the first heat of steel from its new basic oxygen steel plant in Pueblo, Colo. The new 100-tph plant is one of the most modern of its type in the US and is the first to be built in the Rocky Mountain area. Watch for the detailed description of the plant in the September 1961 issue of JOURNAL OF METALS.

Remote gun EB furnace

Reportedly the first electron beam furnace in the US to utilize the remote gun principle for commercial-scale melting of metals was installed recently at the Metals div. of National Research Corp. The furnace, manufactured by the company's subsidiary—NRC Equipment Corp., greatly facilitates the direct melting of loose, unconsolidated feed. Thus far, tantalum and tantalum alloys are the only commercially-available metals; however, company officials expect to produce columbium and other special purpose metals in the near future. The remote gun design is said to avoid costly interruptions due to pressure bursts when melting raw powder, sponge, pellets, or loose scrap. The furnace is also capable of drip melting pressed bars, although this process will probably be avoided in most cases because of the additional pressing requirement. During the furnaces' two-year development, a wide range of metals have been melted, including: tantalum, molybdenum, yttrium, columbium, nickel, iron, and various alloys.

Soviet natural gas

The Intelligence dept. of the British Iron & Steel Federation reports that the Soviet iron and steel industry should receive a big boost from the USSR's ambitious plans to increase its supply of natural gas. Although enormous supplies of natural gas are said to exist in the Soviet Union, inadequate pipelines have hindered the wide-spread use of methane. However, the Soviets plan to add another 16,250 miles of pipeline to augment the existing lines. It is anticipated that by 1965, natural gas will provide 21 pct of the country's total energy capacity, and by 1972 that total is expected to increase to 30 pct. (In the US, natural gas provides for more than 25 pct of the total energy capacity.) Target output for 1965 is 150,000 million cu m compared to the 20,000 million cu m capacity in 1957.

The iron and steel industry in the Soviet Union has been experimenting with natural gas for application in blast and open hearth furnaces. A recent claim that Russian blast and open hearth furnaces are 25 to 30 pct more efficient than US furnaces has been attributed to their successful use of natural gas (See January 1961 issue of JOURNAL METALS, p. 15.) Reportedly the increase in the supply of natural gas will increase furnace production by 1.5 to 2 million tons per year. This year the Russians are expected to inject natural gas in all of the blast furnaces in the Ukraine, and at least 16 other steelworks are known to be using natural gas or plan to do so in the near future.

Sponge iron in Japan

Stora Kopparbergs Bergslags A/B in Sweden has recently concluded licensing agreements with Japanese steelmakers for two Wiberg-Soderfors sponge-iron plants. Hitachi Metal Industries will build a furnace with a nominal capacity of 10,000 metric tons per year. Raw material is expected to be domestic iron sand which after concentration and pelletization will contain 55 pct Fe and some titanium. Tests performed in Sweden on this material have resulted in an iron sponge containing 85 pct Fe with 91 pct reduction. The other firm—Sanyo Special Steel Co. at Himeji—will erect a 30,000 metric ton per year plant to utilize imported high-grade iron ore. Sponge iron produced will be used for the manufacture of ball bearing and other special steels.

Reasons for the installation of these plants are said to be the shortage of scrap in Japan as well as the desirability of obtaining a raw material with minimum amounts of tramp elements for the manufacture of high-grade alloy steels.

Commercial Ti-clad steel

A new development in the production of titanium-clad steel plates was announced recently by Lukens Steel Co., with the production of the first commercial-size order for E. I. DuPont.

Previous cladding methods required the use of an interlayer of vanadium or silver foil between the titanium and the steel, but this is not necessary in the Luken's process. The technique is virtually identical to the roll-bonding procedure which the company uses for other clad steels, such as nickel and stainless. In this procedure, a four-deck pack of two layers of titanium sandwiched between two plates of backing steel is hot rolled, thereby metallurgically bonding the titanium to the steel. The use of a parting compound between the titanium layers permits separation into two clad plates after rolling. Since the titanium and steel surfaces must be kept absolutely clean and free from oxides and nitrides which impair the bond, the titanium-steel pack is purged and flushed with argon during the 6 to 8 hr heating cycle prior to the rolling operation. Bonds produced by this method have attained shear strengths in excess of 20,000 psi.

Electric-furnace spiegeleisen

The New Jersey Zinc Co. recently announced that it has started construction on its new electric furnace project at Palmerton, Pa. which will produce the iron-manganese alloy—spiegeleisen. This new plant is said to be the first commercial installation of this particular smelting system in the U.S. The new installation is reportedly more economical, more efficient, and of wider scope for making various ferroalloys than the present blast furnaces which it will replace. The project, expected to be completed in 1962, is said to be particularly well suited for treating the iron-manganese ores from the company's sterling mine at Ogensburg, N. J.

REPORTER

REPORTER

More direct reduction

Other steel companies are reportedly considering the use of a direct reduction process for producing iron for their steelmaking facilities. Allegheny-Ludlum Steel Corp. has run several tests on the RN direct reduction process in Germany (See July 1961 issue of JOURNAL OF METALS, p. 468.) *American Metal Market* recently reported that Acme Steel Co. is investigating the possibilities for producing directly reduced iron for use in its hot-blast cupola, oxygen converter steelmaking unit. So far, Acme has not decided which process would be used, but indications are that it may be the RN process. Pilot-plant tests have been made using that process at Birmingham, Ala. Lukens Steel Co. is also reportedly running tests on direct reduction processes at its Coatesville, Pa. pilot plant.

Although the cost of directly reduced iron most likely will not differ from the cost of molten metal produced in a blast furnace, the capital investment in a plant for making the directly reduced iron would probably be about one-third that of a comparable blast furnace installation.

In addition to these domestic developments, the first Krupp-Renn iron ore reduction plant to be built in the Western Hemisphere may be constructed in the Republic of Costa Rica. The contracts for the feasibility studies and subsequent construction of the estimated $100-million installation have been entered into by Fried-Krupp of Essen, Germany and International Iron of Costa Rica, Inc. of Houston, Texas. The indicated target of 3 million tons annual production of iron luppen—similar to sponge iron—would make this by far the largest direct reduction plant in the world. The design and construction of the plant will be by the Fried-Krupp Co. for International Iron of Costa Rica, Inc., which is comprised of American, European, and Costa Rican capital. The plant will be located on the Pacific Coast where the deposits owned by International are located.

Cb mill products by Du Pont

Commercial production of mill products of columbium metal and columbium alloys at Du Pont's new Metals Center was announced recently by C. I. Bradford, director of metal products for company's Pigments div. The division has been producing experimental quantities of pure columbium and its alloys since 1955. The new facility, located in the Curtis Bay section of Baltimore, Md., is said to be the first plant designed from the ground up specifically for the production of mill products of refractory metals.

The new installation will produce columbium and columbium alloy sheet, strip, plate, bars, tubes, and other shapes used by metal fabricators. The columbium processed at the new plant is produced from ores by chemical separation methods at the company's Newport, Del. plant. According to company sources, the facility is designed so that tantalum, titanium, and molybdenum products can be produced in the future.

Australian Al complex

The Aluminum Company of America recently announced the establishment of a $100 million aluminum complex in partnership with several Australian corporations —Western Mining Corp., Ltd., North Broken Hill Ltd., and Broken Hill South Ltd. The new concern will be owned 51 pct by Alcoa with the remaining 49 pct held by the Australian companies. The new company—Alcoa of Australia Proprietary Ltd. —plans to begin construction by the end of 1961, and to start operations sometime in 1964. The project will include: bauxite mining facilities and a refining plant in the vicinity of Perth, on Australia's west coast; a 40,000 long ton smelter, fabrication plant, and powder production facilities located 1700 sea miles from the mining site at Geelong, approximately 40 miles from Melbourne.

The Kaiser Aluminum and Chemical Corp. and the Consolidated Zinc Corp., Ltd. of Great Britain are already engaged in an aluminum development project in Australia, and The Reynolds Metal Co., and its affiliate, British Aluminum Co., Ltd. control large bauxite deposits in the northern part of Australia.

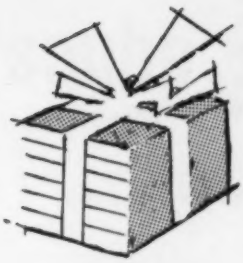
Spray refining

In its 1960 Annual Report recently released, The British Iron and Steel Research Association (BISRA) announced that it has been experimenting with the use of oxygen jets for refining molten blast-furnace metal. The report states that, if a stream of molten metal is atomized by a suitable arrangement of oxygen jets, rapid oxidation takes place.

Batches of hot metal of up to 16 tons have been treated at rates up to 2 tons per min with oxygen rates ranging from 250 to 600 cfm. Pilot-plant tests indicate that at low metal flow rates, the metal can be atomized adequately and the oxygen used very efficiently. Additional experiments are now in progress to determine the form of an atomizing device that will insure adequate utilization of oxygen at high metal flow rates to yield any desired degree of refining. When fully developed, such a spray refining technique should be a simple and inexpensive method for pretreating molten metal to make it suitable for finishing steel in any type of furnace or converter at rates that match the requirements of the finishing process.

Low cost Ti briquets

A new process for producing titanium briquets of at least 90 pct purity at significantly reduced costs has recently been developed by Micro Metals Corp. Using a chemical-mechanical compression process which eliminates the need for expensive titanium sponge normally used with titanium chip to produce briquets, the overall cost of an ingot may be reduced from 5 to 55 pct depending on purity specifications. The company is said to be able to produce 3 lb, all-chip briquets at the rate of 100 to 200 tons per month.



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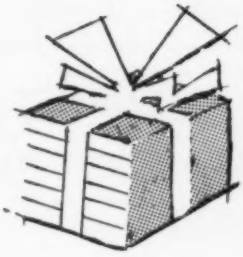

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education

Brooklyn Poly

A metallurgical engineer and one of his students at Polytechnic Institute of Brooklyn have received a research contract for almost $20,000 from the Curtiss-Wright Corp.'s Wright Aeronautical div. for a one year study on the mechanical properties of thin metallic films.

Professor Carmine D'Antonio and Joel Hirschhorn, both members of AIME, are currently concentrating their efforts on testing apparatus and procedure for determining the strength of various materials.

Metallic films are mainly used in miniature electrical and electronic circuits as resistors and conductors. According to Prof. D'Antonio, there is some evidence that these films are extraordinarily strong, and their potential appears very promising.

Hirschhorn, a senior, has assisted Prof. D'Antionio on previous basic and fundamental studies into the mechanical strength of films, and will continue his graduate studies at the Institute specializing in this field.

Engineers in Demand

Although there has been much concern over the tightening market for this year's crop of graduating engineers, a survey conducted by the Engineering Manpower Commission of Engineers Joint Council shows no slackening in the rate at which they are being committed to definite plans following graduation.

A comparison of 49 schools shows 83.8 pct definitely committed (with jobs, entering military service, entering graduate studies, or with other definite plans) as of May 19, 1961. The comparable percentage for 1960 was 84.4 pct. Although there was a 3 pct drop among those with jobs, this was compensated by a 2.5 pct increase in the number going into graduate study, and a 0.9 pct increase in the number going into military service.

These data include only those students for whom the placement offices has information. Results based on a June 16 survey will give a more complete picture.

Copies of the Preliminary Report, *Engineering Graduate Placement Survey*, may be secured from the Engineering Manpower Commission, 29 West 39th St., New York 18, N. Y., at $.25 per copy. The final report will be published later in the summer and will include complete information based on the placement situation as of May 19 and June 16.

University of California

Four members of the College of Engineering at the University of California have been selected as recipients of Miller Research Fellowships. These fellowships make it possible to devote full time to research for a period of one year.

All four of the professors achieving this honor are members of AIME. They are Earl R. Parker and Irving Fatt, July 1961-June 1962; John E. Dorn, July 1962-June 1963; and Jack Washburn, January 1963-December 1963.

• • •

The Department of Mineral Technology at the University of California has reorganized and consolidated its curricula to avoid duplication of courses. This has resulted in two undergraduate curricula, Materials Science which offers courses in metallurgy and ceramic engineering, and Mineral Engineering, with programs in mining, geological engineering, and petroleum engineering.

Graduate programs exist in all of these fields, as well as mineral exploration and engineering science. The latter graduate field emphasizes the scientific aspects of any of the other areas of specialization, and permits graduates in chemistry, physics, and mathematics to work toward advanced degrees without fulfilling all of the requirements of an undergraduate curriculum in engineering.

AUSTRALIA

THE UNIVERSITY OF NEW SOUTH WALES

LECTURER

SCHOOL OF METALLURGY

The University of New South Wales wishes to appoint a Lecturer to the staff of the School of Metallurgy.

The successful applicant will work in the fields of physical metallurgy or metal physics, and should preferably have had formal training and post-graduate experience in the physics of the solid state.

Applicants for this position should have an honours degree or equivalent qualifications in metallurgy or physics.

Salary: £A1772 range £A2477 per annum.

Commencing salary is determined by qualifications and experience.

After passing a medical examination, the successful applicant will be eligible to joint the State superannuation scheme. He will be eligible, after six years of service, for twelve months study leave on full salary.

The University will pay travel expenses for the appointee and his dependent family and will meet reasonable removal expenses for furniture and effects. With the approval of the University and its bankers, married men may be assisted by loans to purchase a home.

Inquiries about the School of Metallurgy may be addressed to the Head of the School, Professor R. H. Myers.

4 copies of applications including the names of 2 referees should be lodged with the Agent-General for New South Wales, 56-57 Strand, London, W.C.2, and a copy forwarded by air-mail to the Appointments Section, Box 1, Post Office, Kensington, N.S.W.

NEW BULLETIN

A new bi-weekly bulletin of "Engineers Available" is now being published by the Engineering Societies Personnel Service, Inc. Distributed free of charge, the bulletin contains synopses of the experience of engineers who have registered with the Service and are seeking a new position.

Any employer interested in receiving the bulletin should so advise E.S.P.S. at 8 W. 40th St., New York 18, N.Y., and your name will be placed on the mailing list. Any engineer who is registered with the Service or wishes to register is entitled to a 35-word notice in the bulletin. Write to the New York office for forms. Your qualifications will be brought to the attention of employers on our mailing list without revealing your identity.

It is planned to extend the publication to the midwestern and western offices in the near future.

La Revue de Metallurgie was created in 1904 by Henry Le Chatelier. In June 1959 it changed format, now appearing in two separate monthly parts.

One part, which preserves the title **Revue de Metallurgie,** is devoted particularly to applied metallurgy, especially the manufacture and working of ferrous and nonferrous metals.

It also includes economic and statistical reports, as well as news of activities of the Societe Francaise de Metallurgie and related institutions.

The second part of the magazine maintains the scientific character of the former publication and, as in the past, includes separate sections devoted to original papers and to abstracts. The title of this part is **Memoires Scientifiques de La Revue de Metallurgie.**

Subscription rates for 1961

Revue de Metallurgie	**$16.50**
Memoires Scientifiques de la Revue de Metallurgie	**$35**
Both publications jointly	**$47**

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▼**Personals:** Please list below your former company and title and your new title and company (or new work) for use in JOURNAL OF METALS. (Copy deadline for personals items is six weeks before date of issue.)

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Any recent activity that would be of interest to members:

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JM

What was Bell Telephone Laboratories doing

ON FRIDAY, JUNE 30, 1961?

It was exploring the communications possibilities of the gaseous optical maser—a device which generates continuous coherent infrared radiation in a narrow beam.

It was preparing an experiment in world-wide communications using "active" satellites powered by the solar battery, a Bell Laboratories invention.

It was completing the development of a new "heavy route" Long Distance microwave system capable of handling over 11,000 two-way conversations at once.

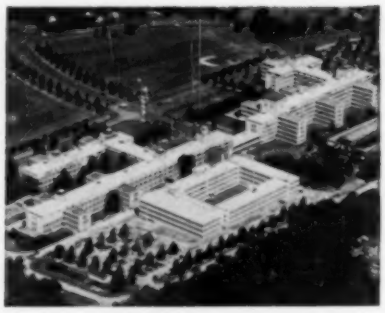
It was developing an anti-missile defense system designed to detect, track, intercept and destroy an enemy ICBM—in a matter of minutes.

It was demonstrating the potentialities of the superconducting compound of niobium and tin for generating, with little power, magnetic fields of great strength.

It was experimenting with an electronic central office at Morris, Ill., which is capable of providing a wide range of new telephone services.

It was perfecting the card dialer which permits, through insertion of a punched card into a slot, automatic dialing of frequently used numbers.

It was developing improved repeaters or "amplifiers" to increase greatly the capacity and economy of undersea telephone cable systems.



It was continuing its endless search for new knowledge under the leadership of scientists and engineers with world-wide reputations in their chosen fields.

Bell Laboratories scientists and engineers work with every art and science that can benefit communications. Their inquiries range from the ocean floor to outer space, from atomic physics to the design of new telephone sets, from the tiny transistor to massive transcontinental radio systems. The goal is constant—ever-improving Bell System communications services.

BELL TELEPHONE LABORATORIES

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BERYLLIUM PROCESSING BY POWDER METALLURGY

This paper describes the powder metallurgical processing of beryllium into fine-grained forms from which finished shapes are made. Emphasis is given to the vacuum-hot-press-machine process which is used for the majority of beryllium parts being made today.

by K. G. Wikle and V. C. Potter

Beryllium is often thought of as a *new* metal important to the Atomic and Space Ages; however, it should also be thought of as the metal used in the largest powder metallurgical operation—with respect to size of parts produced—being carried out today on a commercial basis.

It is usually necessary for the beryllium metallurgist to explain to those not familiar with the metal just why beryllium is fabricated commercially from powders rather than by casting ingots and working these into forged, rolled, or extruded shapes. The chief reasons are as follows:

1) To be of practical use in nuclear, missile, and other applications, beryllium metal must have a fine grain structure, usually of random orientation, uniform density and composition, and must be sound and fabricable by machining and other processes;

2) For beryllium, foundry technology has not been developed which can consistently and competitively produce sound and crack- and porosity-free castings that are either uniformly fine-grained as-cast or can be reliably fabricated by hot- and cold-working processes into fine-grained, machinable shapes having satisfactory mechanical properties;

3) A major breakthrough in beryllium technology occurred in 1946 when C. B. Sawyer and coworkers at the Brush Beryllium Co. developed powder metallurgical techniques (Process Q), which produced beryllium metal having a uniformly sound, dense, and fine-grained structure. Such vacuum hot-pressed metal (QMV) could be machined and otherwise successfully fabricated into shapes having excellent mechanical properties, dimensional stability, and corrosion resistance;

4) By careful control over grain size, grain orientation, and BeO content, the mechanical properties of beryllium powder metallurgical bodies can usually be optimized for given applications. (See Figs. 1 and 2); and

5) Vacuum-hot-press-and-machine is the process by which most beryllium is commercially fabricated into shapes today. This process can be reliably executed to produce beryllium bodies over 5 tons in weight (equivalent in volume to 20 tons and more of steel) and 75-in. in diam and with an excellent uniformity and level of properties. The technology of machining vacuum hot-pressed beryllium metal is highly advanced.

Contrasted with the commercially available and proven vacuum-hot-press-and-machine process, the technology of making large bodies by rolling, forging, drawing, spinning, etc., from either sintered blocks or powder itself has not yet been well developed. Fabricating large bodies by such conventional processes requires large capacity equipment, complicated jacketing, heating, handling, and cooling techniques, involves considerable risk, and leaves something to be desired in final properties, yields, re-utilization of scrap, and overall costs. The art of *powder forging*, however, appears to be advancing at an encouraging rate.

Briefly summarizing, because of the inability to produce sound, fine-grained castings commercially and to fabricate castings successfully into shapes of good quality, the vacuum-hot-press-and-machine process has produced the bulk of all fabricated beryllium used in the US to date.

Quality standards

To realize the need of many of the special handling techniques used in processing of beryllium, it is best to consider here the quality standards established by the beryllium industry itself and required by most customer, government, and society specifications. The goals that must be achieved quality-wise in the final beryllium metal parts are described below for the most popular grade of structural QMV beryllium (S-200) blocks, vacuum hot-pressed from blend powder (P-200):

1) Soundness: density—greater than 99.5 pct of theoretical—and porosity—unconnected and less than ½ pct with no porosity or cracks revealed by dye penetrant or by radiography;

2) Relatively free of high-density inclusions: no inclusions larger than 0.050 in. diam (all included foreign matter is more dense than beryllium radiographically and readily shows up as high density spots);

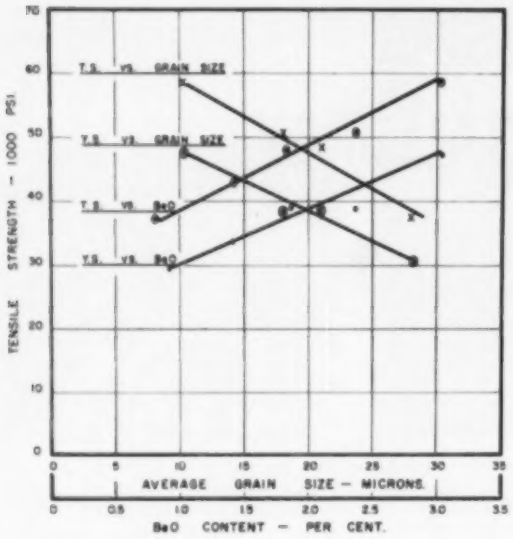
3) Uniform properties;

4) Readily machinable;

5) Uniform, fine-grain size (40 micron avg grain diam or less);

6) Mechanical properties (in any direction): 40,000 psi tensile strength min., 30,000 psi yield strength min., and 1 pct elongation in 2 in. min.; and

K. G. WIKLE and V. C. POTTER are with Brush Beryllium Co., Elmore, Ohio. This paper was presented at the 1961 AIME Annual Meeting in St. Louis.


TENSILE STRENGTH - 1000 PSI
T.S. vs. GRAIN SIZE
T.S. vs. GRAIN SIZE
T.S. vs. BeO
T.S. vs. BeO
AVERAGE GRAIN SIZE - MICRONS.
BeO CONTENT - PER CENT.

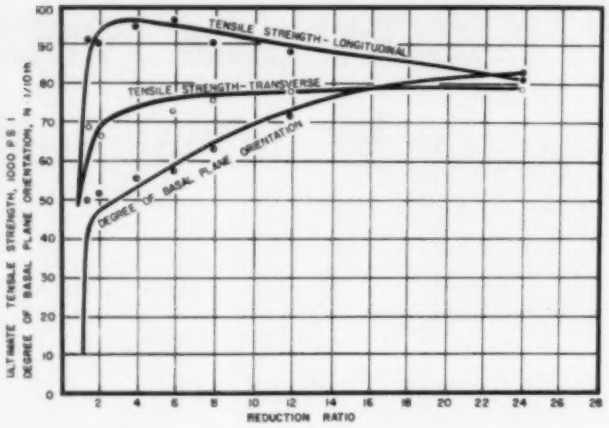

TENSILE STRENGTH - LONGITUDINAL
TENSILE STRENGTH - TRANSVERSE
DEGREE OF BASAL PLANE ORIENTATION
ULTIMATE TENSILE STRENGTH, 1000 P.S.I.
DEGREE OF BASAL PLANE ORIENTATION, IN 1/10th
REDUCTION RATIO

Fig. 1—Left, Effect of grain size and BeO content on the strength of vacuum hot-pressed beryllium. Fig. 2—Above, Effect of reduction ratio on strength and degree of basal plane orientation parallel to rolling plane for QMV Beryllium sheet hot rolled at 1400°F.

7) Chemical composition: 2.00 pct max BeO, 0.18 pct max Fe, 0.15 pct max C, 0.16 pct max Al, 0.12 pct max Si, and 0.08 pct max Mg.

Other considerations

In addition to the need of keeping foreign material out of the beryllium powder which causes observable high density (radiographic) inclusions, beryllium must be *kept out* of the air during its processing. By Atomic Energy Commission health standards, persons shall not be exposed to air containing more than 25 micrograms of Be per cu m without a respirator or fresh air mask. Weighted exposures based on a 3-month period and calculated on a steady 8 hr-a-day work schedule shall not exceed an average of 2 micrograms per cu m. Such exposure limitations impose quite a rigorous need to isolate and ventilate all processes involved with beryllium powder.

In addition to concern about quality and exposure, the beryllium industry is faced with the problem of working with powder presently worth over $60 per lb. Thus, processing must be carried out with a maximum yield so that minimum material is lost completely and minimum scrap must be reprocessed through earlier steps.

Powder processing

A flow sheet describing beryllium powder manufacture and processing at the Brush Beryllium Co. is shown in Fig. 3. The steps involved are discussed below with preliminary reduction and vacuum casting operations also briefly reviewed.

Extraction and Refining of Metallic Beryllium

The first operation in the extractive process for obtaining metallic beryllium is in the reduction furnace where pebbles are formed. The reaction,

$$BeF_2 + Mg \rightarrow Be + MgF_2,$$

yields metallic pebbles in a mixed slag of MgF_2 and BeF_2 called a reduction melt. The reduction melt is crushed, ball-milled, and leached to yield cleaned pebbles of metallic beryllium. The size of the pebbles range from approximately ⅛ to 1½ in. in diam. The chemical assay of these pebbles is in excess of 97 pct metallic beryllium, with BeF_2, MgF_2, MgO, BeO, and other impurities adhering to or entrapped in the pebbles.

Beryllium pebbles are further refined to higher purity by vacuum melting. In this operation, a charge generally composed of 50 pct Be pebbles and 50 pct material returned from machining operations is melted down and poured under vacuum (approximately 500 microns). Melts are poured into graphite molds and vacuum cast ingot is produced weighing approximately 70 lb and measuring approximately 9-in. in diam x 20 in. long. A chemical assay of greater than 98 pct metallic beryllium is achieved. Ingots are meticulously cleaned of mold reaction and surface dross entrapments by chipping and sand blasting.

The ingot produced in vacuum casting is broken up so it can be fed to the attritioning mills for comminution into −200 mesh powder. This is accomplished by a chipping operation.

The top ends of vacuum-cast billets are chucked into a large turret lathe. Then a chipping head, consisting of 10 single point cutting tools and a vacuum pick-up system, advances on the bottom end of the billet. The bottom surface is continuously faced off until the cutting head approaches the chuck. The material (chips) removed by the vacuum pick-up system goes into an *Aerotec* unit where they are removed from the air-stream by cyclone classification and deposited into 55-gal drums. These chips are somewhat similar in shape to those obtained in the machining of cast iron, i.e. curled pieces of approximately 1/16 in. thick, ½ in. wide, and ¾ in. long. Finer products of chipping correspond to coarse powder.

Chip Inspection and Beneficiation

Chips from vacuum-cast ingots (and recycle chips from machining of sintered blocks) are inspected and, if necessary, beneficiated. Two reasons for this operation are to remove large pieces of beryllium and foreign material.

At the inspection station, the chips are conveyed from a hopper past a cross-belt magnetic separator to remove magnetic material and flow onto an inspection tray. The operator visually inspects the chips, removing all large pieces of beryllium and

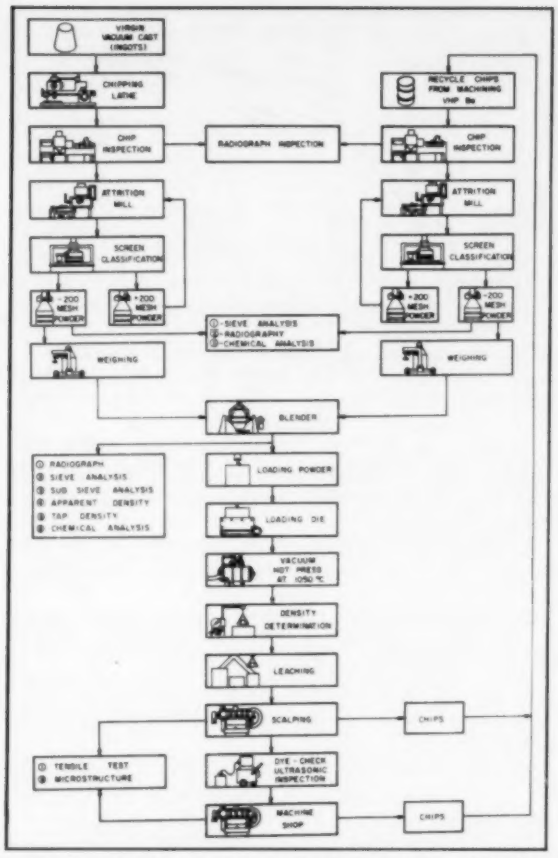


Fig. 3—Flow sheet for powder metallurgical processing of beryllium metal.

visible foreign material. Chips are discharged off the end of the tray over a magnet into steel drums.

A sample from each barrel of inspected chips is radiographically examined. Chips containing significant high-density indications are forwarded to a beneficiation process. Radiographically-clean chips are delivered to the attritioning operation.

In the beneficiation area chips are crushed through a ⅛-in. grate and conveyed past a cross-belt magnetic separator to remove any magnetic material and another sample is removed for radiographic examination and chemical analysis.

Radiographically-clean chips of satisfactory chemical composition are released to the attritioning operation. Chips still containing high-density inclusions are further processed through float-sink and degreasing operations to remove foreign material remaining. Another sample is then radiographically and chemically evaluated. Chips which are found to be clean at this point are forwarded to the attritioning operation and *dirty* chips are returned to the vacuum melting operation.

The inspection and beneficiation of beryllium chips assures that material fed into the attrition process is relatively uniform, contains no large pieces or significant high density inclusions, and the carbon content is acceptably low.

Attritioning

Chips released from inspection and beneficiation are attritioned to —200 mesh powder. Comminution into powder is accomplished by the combination of an attrition mill and 200 mesh screen classification.

The attrition mill consists of two 15-in. diam concave and grooved, water-cooled QMV beryllium plates, one stationary and one rotating at approximately 800 rpm. Chips or oversized particles (+200 mesh) are fed by vibration from an overhead surge hopper through the center of the stationary plate into the cavity between the plates. Here, the oversized particles fall into the grinding zone and by interaction under pressure with the plates and adjacent particles are ground to finer size. Comminuted material exits from the periphery of the plates and is conveyed on to 200 mesh screens of either Rotex or Sweco manufacture. Powder dropping through the screen is collected in a stainless steel drum as —200 mesh product. Material flowing over the screen and into a discharge chute is collected below in a separate drum as oversize material. Oversize material is recycled through the attrition mill until comminuted into —200 mesh powder.

The attrition mills are driven by 20 hp motors consuming approximately 1.5 (fine powder) to 3.5 (coarse chips) kw-hr per lb of —200 mesh powder produced. The production rate averages about 16 to 18 lb of —200 mesh powder per hr and plates grind in excess of 7000 lb of —200 mesh product per lb of plate wear. The screen classification approaches 95 pct efficiency in separating —200 mesh powder from oversize material.

By means of a continuous sampling system representative powder from each lot (barrel holding 350 lb) undergoes chemical, radiographic, and sieve analysis. These analyses are for process control and provide information by which proper blends can actually be made for the vacuum hot-pressing operation.

Blending Operations

Providing the vacuum hot-pressing process with columns of uniformly blended powder is an important factor in producing dense, uniform blocks free of voids, cracks, and porosity. Since uniformity of QMV beryllium is carefully evaluated by density determination, metallographic examination, chemical analysis, tensile testing and ultrasonic, dye-penetrant, and zy-glo inspection, blending of input powders must be carried out as effectively as possible. Segregation during subsequent loading of dies and their transport into hot-pressing furnaces must also be avoided by careful handling.

In the case of small blocks, blending may consist of merely mixing only one lot uniformly. However, most blocks processed require blends of one or more lots of both virgin and recycle powder. For these blends, several powder lots are placed in large blenders and blended for various periods of time, depending on the weight and the blender used. Present blenders range from 10 to 100 cu ft (300 to 3000 lb.) capacities in double-cone and twin-shell designs.

Six specific reasons for blending beryllium powders going into the vacuum hot-pressing process are as follows:

1) To insure a uniform, homogeneous vacuum hot-pressed block,

2) To adjust chemical composition to desired levels,

3) To adjust powder particle size and particle size distribution to desired levels,

4) To combine powder from recycled machine chips with that from vacuum cast ingots,

5) To combine or produce alloy powders which can be vacuum hot-pressed into blocks having a uniform and homogeneous structure and analysis, and

6) To avoid segregation of fines (or coarse) particles in planar and in irregular volumes which may cause actual or dye-penetrant-determined cracks and miscellaneous porosity and voids, respectively.

Loading Powder

The loading of blended powder into sintering cans must meet three requirements. The first requirement is to deposit powder in a steel can in such a manner that the column formed is completely uniform and homogeneous with no segregation or fissures. The second is to prevent the inclusion of high-density particles which will be the basis of radiographic rejection. The third is to provide a representive sample of the powder for chemical, radiographic, and sieve analysis.

The powder column should be as dense as possible to minimize the amount of entrapped air which must be removed during subsequent vacuum hot-pressing and to provide the shortest possible powder column so that a minimum of consolidation is necessary during vacuum hot-pressing. An excess amount of air in the powder column increases the possibility of a powder *blow* occurring during evacuation and associated segregation. The first two requirements are met by attaching the steel can in which the powder is loaded to a vibrating table.

The powder flows from the blender into a vibratory feeder and into the steel cans, all of which are enclosed in a clean, ventilated compartment. Inclusion of objectionable high-density matter is avoided by thoroughly pre-cleaning the steel cans.

Continuous-type samples of blended powders are collected as the steel can is being filled. These samples are evaluated radiographically, chemically, and for average particle size and distribution. Chemical and radiographic analysis essentially guarantee that the resultant vacuum hot-pressed block will comply with necessary requirements. Although particle size specifications require only that the powder be −200 mesh, maintenance of uniform vacuum hot-pressed properties dictate that the average particle size and distribution be controlled. The analysis range of typical −200 mesh powders vacuum hot-pressed at the Brush Beryllium Co. is as follows:

Analysis Range of Typical −200 Mesh P-200 Beryllium Powder

Sieve Analysis (Schuman Interpretation)	Avg Particle Size	
	Diam	Modulus
Min. Size	28μ	0.8 to 1.2
Max Size	42μ	1.8 to 2.2
Avg Fisher Sub-Sieve Particle Diam		
Min. Size	13μ	
Max Size	16μ	
Apparent Density: 0.6-0.7 g/cu cm		
Tap Density: 0.98 to 1.02 g/ cu cm		
Moisture Content: 0.3-0.4 pct		

Vacuum Hot-Pressing

In this operation homogeneous columns of beryllium powder contained in steel cans are consolidated into uniform, dense, fine-grained blocks. Eight separate steps generally used in the vacuum hot-pressing operation are as follows:

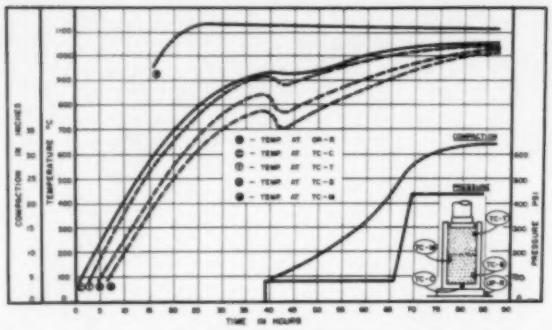


Fig. 4—Time-Temperature, -Pressure, and -Compaction relations in vacuum hot-pressing a 39½-in. diam x 38½-in. high QMV beryllium block.

1) Loading a steel can containing uniformly-blended and vibration-packed (approximately 55 pct density) powder into a steel, graphite, or carbon hot-pressing die;

2) Loading this die with required compaction tooling into the furnace and sealing the furnace vacuum tight;

3) Evacuating the furnace to about 500 microns;

4) Heating so that a powder temperature of to 1000° to 1100°C is attained;

5) Applying 200 to 600 psi to compact the vibration-packed powder into a dense body of essentially 100 pct density;

6) Unloading the compacted block, die, and tooling from the furnace and placing in a special cooling chamber;

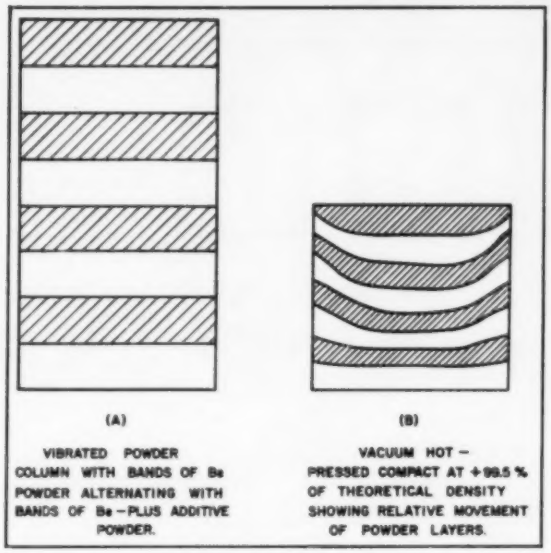
7) Unloading the vacuum hot-pressed block from the die; and

8) Stripping the beryllium block from the steel can.

To illustrate the temperature, compaction, and pressure relationships with time in making a typically-sized QMV vacuum hot-pressed block (39½ in. diam x 38½ in. high), the data in Fig. 4 is presented. Here three thermocouples were placed in outer surfaces of the powder column at locations TC-T, TC-M, and TC-B. These locations were all inside the steel can adjacent to the can surface with TC-T approximately 3 in. from the bottom, TC-M at the site of the finished height of the compact, and TC-T approximately 3 in. below the top of the powder column. TC-C was at the bottom center and outside the steel can.

In Fig. 4 time is plotted on the abscissa and temperature, pressure, and compaction are plotted on the ordinate. This graph indicates that the powder in the center of the compact requires more heating time (20 hr) to arrive at the final pressing temperature and for most of the run is >100°C colder. The temperature of the bottom of the powder column is generally between that of the center and the top of the column. Notice the drop in temperature and the slow recovery when the pressure is initially applied. This is the result of the center portion of the compact rapidly absorbing heat from the outside because of the increased heat conduction of the denser powder.

In vacuum hot-pressing, effecting suitable compaction is essentially a matter of heating all of the powder column uniformly into the range of 1000° to 1100°C. At this temperature consolidation close to 100 pct of theoretical density is possible, if a


(A)
VIBRATED POWDER COLUMN WITH BANDS OF Be POWDER ALTERNATING WITH BANDS OF Be – PLUS ADDITIVE POWDER.
(B)
VACUUM HOT – PRESSED COMPACT AT +99.5 % OF THEORETICAL DENSITY SHOWING RELATIVE MOVEMENT OF POWDER LAYERS.

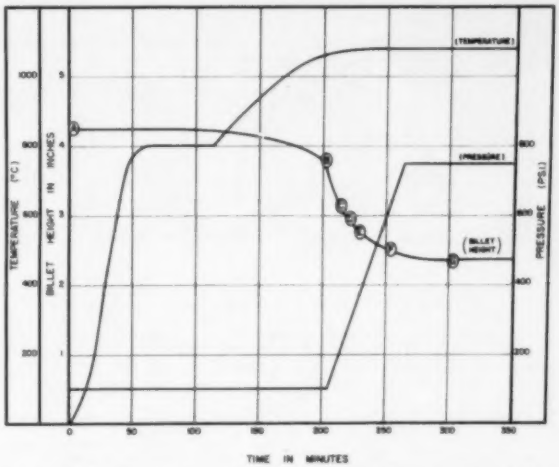


Fig. 5—Left, Illustration of relative material movement during vacuum hot-pressing of beryllium powder. Fig. 6—Above, Temperature pressure, compaction, and density relationship with time during vacuum hot-pressing 2½-in. diam x 4¼-in. high beryllium powder column.

uniformly packed powder column is provided, and if length-to-diameter ratio and wall friction do not cause too much variation in *effective* pressure throughout the column. Since this was the case with the pressing described in Fig. 4, compaction to a final density of greater than 99.5 pct density was achieved when all parts of the powder column arrived at a minimum temperature of 1000°C.

Vacuum hot-pressing is carried out in several types of furnaces at the Brush Beryllium Co. Furnaces installed in the late 40's and early 50's were simple, single-retort, gas-fired units capable of pressing cylindrical and rectangular blocks as well as slabs from about 10 to 300 lb. In 1955, the world's largest vacuum hot-pressing unit was completed and employed in producing blocks of about 3000 lb for reactor parts. In 1960, the largest beryllium as well as the largest powder metallurgical body—75 in. diam weighing nearly 5 tons—was produced in the hot-pressing unit. This furnace, because of its large size, is electrically heated with resistance windings whose temperature can be closely controlled. The furnace may draw up to 450 kw during heat-up; it sits in a large 1000-ton press which is disassembled when the pressing assemblies are loaded and unloaded. Five other smaller furnaces of this resistance-heated, double-retort type were built and put into productive use from 1957 through 1960. Two units of this type are used for pressing up to 40 in. QMV beryllium blocks. Other smaller vacuum hot-pressing units are heated by 150-200 kw, 3000 cycle and 180 cycle induction coils.

Induction-type furnaces consist of single retorts and are simple to construct, operate, and maintain. Unfortunately, pressing die size (graphite or carbon) and induction coil and inductor design limit the maximum-sized block that can be pressed; so, these units produce small-to intermediate-sized QMV blocks. High and low frequency generating equipment cause induction units to be more expensive than resistance-wound and gas-fired furnaces. In 1961, 14 vacuum hot-pressing units are available at the Brush Beryllium Co. to press QMV beryllium blocks weighing from a few to over 11,000 lbs.

The vacuum hot-pressing process is possible with powdered beryllium metal because of its atomic mobility and plasticity at 950° to 1100°C which permits a high degree of densification to be brought about by evaporation-condensation, surface diffusion, volume diffusion, and plastic flow mechanisms. Generally speaking, the purer and cleaner the beryllium particles are, the easier the vacuum hot-pressing process can compact powder columns to high density. However, during the processing of ingots into powder and then into compacts, the beryllium develops tremendous surface areas which react with oxygen to form an oxidized exterior. At lower temperatures—below about 850°C—this oxidation is a continuous film, but this film breaks down sufficiently into a granular-type of oxide before 950°C is reached which permits sintering to occur. Increasing oxide contents do cause increasing difficulties to the vacuum hot-pressing process; however, it appears that other impurities which accompany higher oxide-containing (recycle) powders compensate for the additional *refractory* BeO blockading by significantly catalyzing the sintering process. The beneficial effect of these impurities seems to balance out the interparticle blocking and structural strength contributions of the BeO at least up to a 2 pct content.

Some of the compaction problems encountered in vacuum hot-pressing, are best illustrated by the results of two laboratory investigations. Fig. 5 shows the material flow which occurred during pressing a vibrated column of beryllium powder, 2½-in. diam x 4¼-in. tall. This column was made up of −200 mesh powder layers of beryllium and of beryllium plus 1 wt pct metallic additive. This additive is similar in vacuum-hot-pressability, particle size, and density to beryllium. Preliminary tests carried out proved that the alternate powder layers remained uniform in height, horizontally, and did not blend together during vibratory-packing of the die. Fig. 5(A) shows the column before vacuum hot-pressing and Fig. 5(B) after densification in vacuum at 1050°C using 750 psi compacting pressure on one movable punch. The cross-section developed on the actual sample by macro-etching

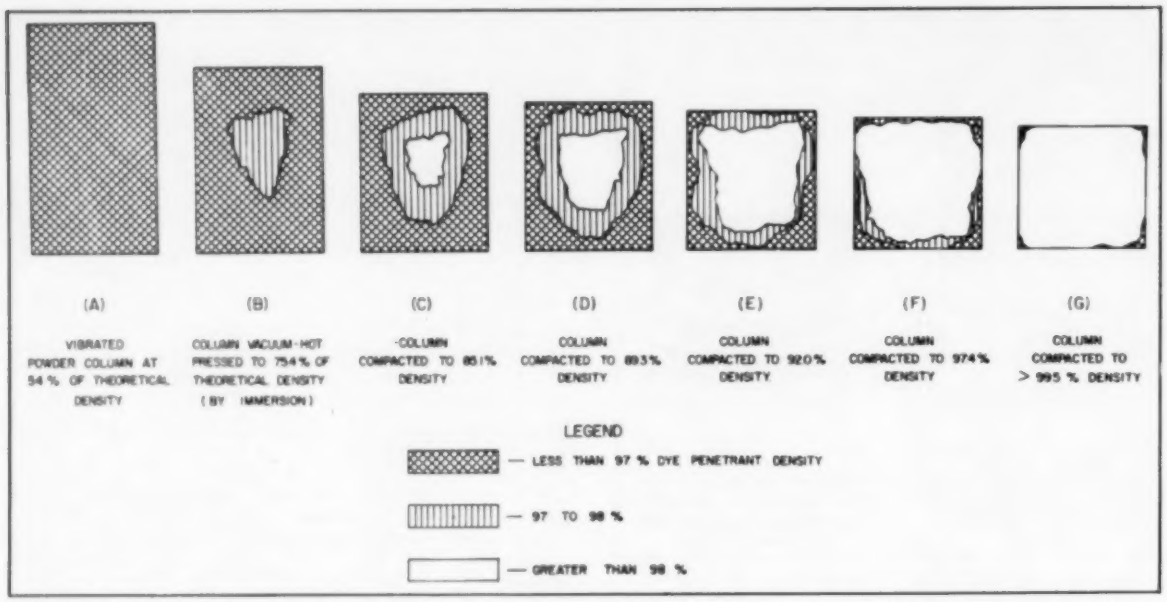

(A)
VIBRATED POWDER COLUMN AT 54 % OF THEORETICAL DENSITY
(B)
COLUMN VACUUM-HOT PRESSED TO 754 % OF THEORETICAL DENSITY (BY IMMERSION)
(C)
COLUMN COMPACTED TO 851% DENSITY
(D)
COLUMN COMPACTED TO 893% DENSITY
(E)
COLUMN COMPACTED TO 920% DENSITY
(F)
COLUMN COMPACTED TO 974% DENSITY
(G)
COLUMN COMPACTED TO > 995 % DENSITY
LEGEND
— LESS THAN 97 % DYE PENETRANT DENSITY
— 97 TO 98 %
— GREATER THAN 98 %

Fig. 7—Illustration of dye-penetrant porosity elimination during vacuum hot-pressing of 2½-in. diam x 4¼-in. high column of —200 mesh QMV beryllium powder.

technique shows that material flow in vacuum hot-pressing is similar to that which takes place in cold pressing. The essential features of compaction by vacuum hot-pressing as demonstrated by this study are as follows:

1) The central body of the powder is first more highly densified. This is because an uncompressed volume of powder is immediately produced just in front of the punch because of lateral flow restrictions and *bridging* of material, and wall friction retards compaction along the lower wall surfaces. As a result of this early central-body compaction, the layers bend down in this section.

2) After considerable compaction of the central body volume, the zone of maximum densification spreads to the upper corners, to the central wall areas, to the area just below the punch, and finally, to the bottom corners, in that order. The extreme surfaces are not compacted to greater than 99.5 pct of the theoretical density; in fact, these areas cause the compact inevitably to be somewhat less than 100 pct dense. Lateral flow restrictions at the surface of the punch and die bottom and wall friction at the walls prohibit full surface densification.

3) Material plastically flows *laterally in* from the peripherial areas at the top in order to densify the convex area just below the punch. Material plastically flows *laterally out* from the central areas at the bottom in order to densify the bottom corners. This radial movement inward at the top and outward at the bottom explains the curved shape and uneven thickness of the layers after hot-pressing.

4) In this investigation only one movable punch was used. If two movable punches had been employed, a condition identical to that of two pressings of Fig. 5(B) placed bottom to bottom would have resulted.

5) Reaching uniform high densities in vacuum hot-pressing with one movable punch depends chiefly upon the following: sufficient pressure, time, and temperature to get areas directly in front of the punch, along the wall, and in the bottom corners up to densities approaching that of the central body; and a minimum of wall friction resulting from smooth or lubricated walls or from sufficiently small length to diameter ratios and/or volume-to-surface ratios.

Figs. 6 and 7 further demonstrate conditions existing in the vacuum hot-pressed beryllium compact as it is pressed up to greater than 99.5 pct of theoretical density using one movable punch. At point "A" on Figs. 6 and 7 the —200 mesh powder was packed up to 54 pct by vibration. In (B) through (G), the 2½-in. diam x 4¼-in. long powder column was pressed up to greater than 99.5 pct of theoretical density in a laboratory furnace. Pressings at various stages of compactions were sectioned, polished, etched, and examined with dye penetrant to locate porous areas. The dye penetrant technique had previously been carefully calibrated to show that dark red *bleed out* occurred in the less than 97 pct density areas, pink *bleed out* in 97 to 98 pct dense areas, and no *bleed out* in areas more than 98 pct dense. The porosity found in the studies illustrated in Fig. 6 showed, as in the previously-described study, that the central body first compacts more densely in hot-pressing; this area of maximum densification extends to the top corners, the side walls, the top center, and then to the bottom corners. During the last stages of densification to greater than 99.5 pct of theoretical density, the surface areas must be densified against the resistance of vertical wall and lateral punch and die face frictions. This final densification is the most difficult task to accomplish in vacuum hot-pressing beryllium because of temperature and pressure limitations imposed by die material and possible metal-die reactions.

Density Determination

The density of all QMV blocks is determined after stripping from the steel can and cooling to room temperature. The density is then converted to pct of theoretical density. The density is determined by the water displacement method with corrections

Table I. Potential Processes Other Than VHP-Machine for Fabricating Beryllium Using Powder as a Starting Material

Vacuum Hot-Press and Work Processes

Process	Brief Description	Application	Advantages	Disadvantages
VHP and Warm Extrude	VHP-Machined billets bare extruded at 800°-1350°F.	Small diam rod and shapes	Sm. Eq., Imp. Props., Thin Shapes, Fur. Form., G. Yd., G. Dim. C., Tech. W. D.	Tl. Exp., Sc. Rem., P. Lim., L. Prod., Low Tr.
VHP and Hot Extrude	VHP-Machined billets jacketed in steel and extruded at 1650°-2000°F and dejacketed.	Long bar, rod tubing and simple shapes	Imp. Props., Lg. Shapes, H. Prod.	Lg. Eq., J. and Dj., Sc. Rem., Low Tr., P. Lim.
VHP and Roll	VHP-Machined blocks jacketed and hot rolled.	Plate, sheet, strip and foil	Thin Shapes, Lg. Shapes, Imp. Props., Fur. Form., G. Dim. C., G. Yd.	J. and Dj., Lg. Eq., Sc. Rem., Low Tr. (short), P. Lim.
VHP and Warm Forge	VHP-Machined billets are forged at 800°-1350°F.	Small simple shapes	Imp. Props., H. Prod.	Tech. N. W. D., Dif. Contr., Tl. Exp., Sc. Rem.
VHP and Hot Forge	VHP-Machined billets jacketed and forged at 1500°-2000°F.	Large simple shapes	Lg. Shapes	Tl. Exp., J. and Dj., Lg. Eq.
VHP and Form	VHP-Machined plates draw formed at 800°-1350°F.	Formed plate	G. Yd., Imp. Props., H. Prod.	Tech. N. W. D., Dif. Contr., Tl. Exp., Sc. Rem.
VHP and Draw	VHP-Machined and extruded rod and tube further drawn at 800°-1350°F.	Wire tubing	Thin Shapes, Imp. Props., G. Dim. C.	Tl. Exp., L. Prod., Sc. Rem., P. Lim.
VHP, Roll, Form and Join	Sheet hot formed, spun drawn into metal parts 800°-1500°F. Joined by rivetting, bolting, brazing, welding and bonding.	Thin or large structures	Thin Shapes, G. Yd., Imp. Props., Lg. Shapes	Tech. N. W. D., Lg. Eq., Low Tr. (short), Sp. Eq.
Direct Powder Forming Processes				
Powder Forge	Jacket powder, cold or warm pre-press and forge in closed dies 1650°-2000°F.	Large, intermediate and small parts of relatively simple shape	H. Prod., G. Yd., Lg. Shapes, No Vac.	Tl. Exp., Lg. Eq., J. and Dj., Dif. Contr., Cont. Prob.
Hot Press in Air	Press in graphite or Hi-Temp dies at 2000°F in air.	Cylindrical or rectangular bodies	No. Vac., H. Prod.	Tech. N. W. D., Tl. Exp., Dif. Contr., Cont. Prob., Q. Prob.
Cold Press—Sinter	Compact powder at 25-75 TSI, sinter in <5μ vacuum at 1175°-1225°C. Coin if necessary.	Small simple parts in large quantities	H. Prod., G. Yd., Sm. Eq.	Tl. Exp., Dif. Contr., Sp. Eq., Q. Prob.
Warm Press—Extrude, Roll or Forge	Compact powder with 25-75 TSI at 800°-1350°F and then extrude, roll or forge.	Extrusions, sheet and forgings	No Vac., Hi. Prod., Imp. Props., Sm. Eq., G. Yd.	Dif. Contr. (see rolling extrusion and forging above)
Pressureless Sinter	Pack virgin powder into graphite die. Sinter in <5μ vacuum at 1175°-1225°C to approx 95 pct density.	Simple and special shapes, preforms for further working	Sm. Eq., Fur. Form.	Dif. Contr., Q. Prob., Must use virgin powder.
Pressureless Sinter and Work	Pressureless sintered billet of 95 pct density is further worked by extrusion, rolling or forging.	Extrusions, sheet and forgings	Sm. Eq., Fur. Form.	Dif. Contr., Q. Prob., Must use virgin powder, (see rolling, extrusion and forging above)
Hot upset sheet	Cold press, hot press and hot upset by forging at 1650°-2000°F.	Plate and sheet with "three dimensional ductility"	Imp. Props., G. Yd.	Lg. Eq., L. Prod., P. Lim., J. and Dj., P. Dim. C., H. C.

Legend of Abbreviations

Advantages		Disadvantages	
H. Prod.	— Highly Productive	L. Prod.	— Low Productivity
G. Yd.	— Good Yield	Lg. Eq.	— Large equipment necessary
Lg. Shapes	— Large or long shapes can be produced	Low Tr.	— Low transverse properties
Sm. Eq.	— Only small equipment necessary	P. Dim. C.	— Poor dimensional control possible
Imp. Props.	— Improved strength and/or ductility results	Tech. N. W. D.	— Technology not well developed
Fur. Form.	— Product can be further formed	Spec. Eq.	— Special equipment necessary
G. Dim. C.	— Good dimensional control possible	Dif. Contr.	— Difficult to control process
Tech. W. D.	— Technology well developed	Cont. Prob.	— Contamination Problem
No Vac.	— No vacuum furnace necessary	Q. Prob.	— Quality problem or product no yet accepted by trade
Thin Shapes	— Thin sheet or wire can be produced by this technique	Tl. Exp.	— Tooling expensive
		J. and Dj.	— Jacketing and de-jacketing necessary
		H. C.	— High Cost
		Sc. Rem.	— Scrap must be remelted as opposed to just re-attritioning
		P. Lim.	— Process limited in scope

for the temperature of the block and water utilized in the calculations. Theoretical density of vacuum hot-pressed blocks is obtained by correcting the density of pure beryllium (1.8477 g per cu cm) for the BeO content (3.025 g per cu cm) of the block.

Special scales are used to obtain the dry and wet weight of the blocks accurate and readable to 0.01 pct permitting density determinations to the fourth significant figure (1.851 g per cu cm) and calculations of per cent of theoretical density to greater than three significant figures (99.7 pct).

A 99.5 or greater pct of theoretical density is required for processing QMV beryllium into parts. Those blocks with less than 99.5 pct are either repressed or remelted. This is the first of many tests in the quality evaluation program of QMV beryllium.

Surface Preparation

The vacuum hot-pressed blocks are leached after the density determination has been made and approval is given for further processing.

After density determination the vacuum hot-pressed blocks are leached 8 hr in sulfuric acid. Leaching removes the outer surface in which beryllium is alloyed with iron, and cleans the surface of oxidized areas and other inclusions adhering to the surface of the block. The advantage of this leaching operation is a clean surface which allows chips obtained during scalping to be attritioned directly into powder with a minimum of beneficiation.

Scalping removes the outer surface skin and machines the pressed blocks to standard stock sizes. Milling machines, lathes, or vertical boring mills are used, depending upon the size and shape of the block. All machines are equipped with vacuum chip pick-up units which insure low atmospheric dust counts and efficiently collect chips with minimum contamination.

After scalping, blocks have smooth and parallel surfaces and square corners and are then suitable for effective evaluation by dye-penetrant and ultrasonic inspection.

Inspection and Final Machining

After the scalping operation, all vacuum hot-pressed QMV blocks are examined by ultrasonic and dye-penetrant inspection. These procedures not only reveal defects that would be objectionable in final parts, but also indicate processing difficulties so that corrective action can be initiated immediately. Location of defects minimizes subsequent machining expenses, allows maximum use of material, and insures maximum metallurgical quality in final parts.

The final machining operations are carried out with a variety of machines which depend on the size and shape of the QMV block, the number, size and shape of final parts, and the final dimensional tolerances required. Special machines are not necessarily used but all are equipped with vacuum chip pick-ups and suitable ventilation. Test bars are removed during final machining for tensile property and grain structure evaluation. Various inspections are carried out between machining operations.

As required, all finished parts are inspected with the standard techniques: dye-penetrant (or Zyglo), radiographic, dimensional, and tensile properties. For special applications additional inspection and evaluation may be conducted.

Advantages

The advantages of the vacuum-hot-press-and-machine process for beryllium can be listed as follows:

1) Beryllium metal can be readily ground into powder of good chemical purity;
2) Beryllium powder can be readily and consistently compacted in vacuum with low pressures at 1050°C into uniformly dense bodies of over 99.5 pct of theoretical density;
3) Grain structure produced is fine (less than 40 microns with −200 mesh powder) and randomly oriented;
4) Mechanical properties are relatively good in all directions and in all parts of pressings;
5) Strength can be improved by using recycle powders high in BeO content;
6) By use of fine powder (sub-sieve), strength, stability, and other properties can be improved;
7) Excellent machinability and machining technology is highly advanced so that fabrication by this method is relatively efficient;
8) Chips generated by machining (dry) can be returned directly to the powder attritioning process and are then immediately reusable;
9 Vacuum hot-pressed beryllium can be readily rolled, forged, and extruded using standard techniques developed for beryllium;
10) Commercial equipment is available to press bodies up to 75-in. diam and up to about 6 tons in weight; and
11) Alloys, if desired, can easily be made from mixed powders with minimum segregation.

Other fabrication processes

This paper primarily described the powder metallurgical steps involved in the vacuum-hot-press-machine process which has been and is being used for the bulk of beryllium metal parts being made today. However, other processes which have possible commercial application in fabricating beryllium should be briefly mentioned. At the moment it would appear that all processes having good commercial potential involve beryllium powder as a starting material. Powder must be used in order to guarantee fine-grained, sound bodies having optimum mechanical properties and capable of being readily machined.

Table I lists eight processes which are being used or have been employed at one time or another to convert vacuum hot-pressed beryllium into more highly fabricated shapes. Also are listed six methods of consolidating beryllium powder into shapes other than the basic vacuum hot-pressing process. Advantages and disadvantages for each technique are pointed out.

Warm and hot extrusion, hot rolling, and hot forging of vacuum hot-pressed beryllium billets and blocks are processes readily carried on, provided practices established for beryllium are used. Warm extruded rod and tubing is further drawn at elevated temperatures into wire and *cold* finished tubing. Beryllium sheet metal is readily hot bent, drawn, spun, and formed into shapes which can be joined together by rivets, bolts, soldering, brazing, welding, and adhesive bonding.

Direct powder techniques, such as powder forging, warm press-hot extrude, pressureless sinterwork, and hot upset forging of sheet, are currently being used to fabricate special products. Powder forging is, by necessity, a method of making parts in the larger hydraulic presses. The British, at one time, found pressureless-sintering a convenient way of making extrusion billets for tubing from *virgin* powder. This process did not require the large capital investment necessary for vacuum hot-pressing facilities; however, lack of uniformity in sintering from powder lot to powder lot and in various parts of the sintered body made yields lower than desired. Hot-upset-forging is a technique of shaping sheet from powder which results in a minimum of directionality but a high strength level. Processes which avoid a lengthy vacuum sintering period, fully densify the powder as well as involve some degree of plastic deformation appear to develop good strength and ductility combinations in bodies fabricated from beryllium powder.

GASEOUS REDUCTION OF OXYGEN-CONTAINING COPPER

For centuries, wood poling has been used for reduction of oxygen-containing copper in the smelting operation. Substitutes have been tried, but apparently without complete commercial success until the development of the gaseous reduction process described in this paper.

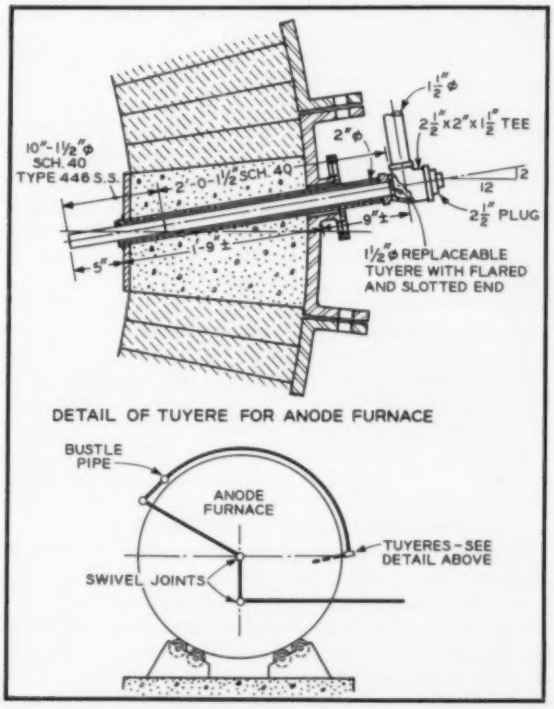
by Leonard Klein

The art of fire refining of copper, consisting of melting, oxidation, slag removal, and finally reduction with wood poles, has been practiced for many centuries. An authentic record, discussed in detail in De Re Metallica, mentions that as early as 1200 AD, workers in this field of metallurgy were already acquainted with, and practiced, the basic principles of copper purification. Undoubtedly, the art was well developed many centuries before the first written records began to appear. Notwithstanding the many advantages gained by application of mechanical contrivances and by the tremendous increase in size of operations over the centuries, the reduction phase of the process has persisted essentially the same to the present time except for a very recent development.

Numerous articles have been published and many patents have been issued on the subject of substitutes for wood poling in the past half century. They have dealt with a variety of gaseous, solid, and liquid reducing agents. But as far as is known, no procedure has ever been pursued to a complete success on a large operating-plant scale with the single exception of gaseous reduction of oxygen-containing copper revealed in detail in this paper. This gaseous reduction process is the result of the concerted efforts of C. R. Kuzell, M. G. Fowler, John H. Davis, and the author. US Patent No. 2,989,397 has been granted to the author and his co-workers for the process.

The natural gas which has become available to smelters in the southwestern United States consists of CH_4, CO_2, and N_2 with small amounts of higher homologues of CH_4 up to pentane, C_5H_{12}. It was found that when this natural gas is passed into a bath of molten, oxidized copper, the reducing effect is small and intolerably slow. However, when the natural gas is reformed to hydrogen and carbon monoxide, and these gases are passed into a bath of molten, oxidized copper, then the reducing effect is potent and quite rapid.

A natural gas-air reformer with ample capacity for plant experimentation become available at the Douglas reduction works of Phelps Dodge Corp., at Douglas, Ariz., in 1958; thus, plans were formulated, the necessary auxiliary equipment designed and installed, and experimentation initiated in the gaseous reduction of oxygen-containing molten copper for the production of anode-quality refined copper in two 13-ft diam x 20 ft long, cylindrical, rotating, gas-fired furnaces. The reduction of the first charge of over 100 tons was encouraging. Pipe lances, initially used for introducing the reducing gas into the bath, were soon replaced by tuyeres. In the course of time, a larger reformer was designed and built locally, more complete auxiliary equipment was added, instrumentation was modified and augmented, and full automation of the reformer



Detail of tuyere for anode furnace.

LEONARD KLEIN is research chemist of the Phelps Dodge Corp., Douglas, Ariz. This is a summary of a paper presented at the 1961 AIME Annual Meeting.

plant became possible, thus eliminating all operating attendants.

Anode furnace refining

Molten copper, as received from the converter, normally contains a variety of impurities, mainly sulfur, oxygen, and iron. The state of oxidation of the copper when transferred varies considerably, depending on local practices and requirements. It may be low or high *blister* in quality. In the former case, much more oxidation is required in the anode furnace than if the bulk of the oxidation had been previously carried out in the converter. The state of oxidation of the charge is readily determined by the use of the so-called *say-ladle* samples.

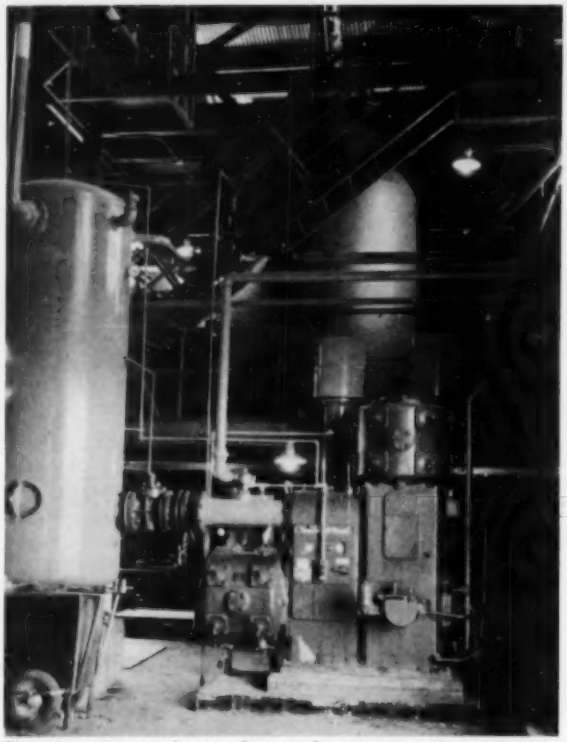
After the anode-furnace operator has attained his preferred endpoint in oxidation, there is normally more or less of the so-called supernatant *oxide* slag to be removed. This is skimmed from the surface of the bath into a ladle and returned to the converter. It is important to skim thoroughly in order to avoid retention of oxidized material, which subsequently extends unnecessarily the volume of reducing gas and treatment time required for reduction of the molten oxidized copper.

At this point the copper has been dispossessed of the bulk of its original impurities except oxygen, the latter having been augmented in the meantime to as much as 0.6 to 0.7 pct. This undesirably large content of oxygen must be diminished by a reducing agent, heretofore poles, now reformed gas.

As soon as the skimming is terminated, the reformer plant is activated, reformed gas is directed through the upturned tuyeres and vented from the furnace ports to the atmosphere. As the furnace is rotated, the two tuyeres are slowly submerged, the bubbling of the gas through the bath becomes audible, and the turbulence of the bath becomes observable through the poling door. The degree of submergence varies according to the judgment of the operator.

If one or both tuyeres should start plugging during the cycles of refining, the tuyeres are elevated above the bath, the air or gas, as the case may be, is vented to the atmosphere; the tuyeres are barred or drilled out as needed, and the furnace is returned to blowing position. Ordinarily, cleaning out the tuyeres is a matter of less than 5 min. The plugging of tuyeres is quite erratic. Many charges are processed without any plugging, and some charges require attention one or more times, particularly during the reduction cycle. Normally, the operating back pressure in the furnace line varies from 15 to 20 psi. When the tuyeres start to plug up, the gauge pressure rises, and when, under abnormal conditions, it approaches 25 psi, it is considered advisable to turn up the furnace and clean tuyeres.

Reduction is continued until the oxygen content has been lowered to a predetermined point satisfactory for successful casting and specifications. The oxygen content is normally decreased to a point below 0.2 and at times to 0.03 to 0.05 pct. It has been found advantageous to carry the reduction of oxygen to a somewhat lower content than is actually required for casting and specifications, since there is a pick-up of oxygen in the time interval between completion of reduction and actual sampling of the copper later as it overflows the tilting spoon into the molds. This pick-up has been found to approach 0.1 pct. The sample taken after casting has been started is the one used officially for analysis.



The Morenci natural-gas reformer plant—center, air compressor; left, reformer; and center background, catalyst chamber.

Comparative costs

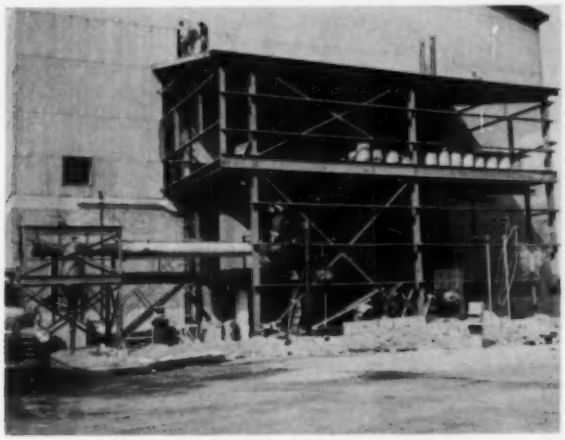
Before presenting a comparison of relative costs of reduction with wood poles vs reformed gas, it will be informative to call attention to two methods of calculating basic costs. Generally, costs are determined by using as a divisor the tons of salable anodes produced. However, as every copper metallurgist knows, the actual tonnage of copper refined is always greater than that of salable anodes. The anode tonnage values have been used herein to arrive at comparative amounts of the two reducing reagents.

Again, there arises the question of average basic prices of wood poles, or natural gas and of electrical power at the various plants producing anodes. The following estimated average prices have been assumed herein for the purposes of discussion:

Wood Poles: The price of green oak poles varies chiefly with distance of plant from the forest. The value of 1.5 cents per lb poles fob anode furnace floor will be used as a representative cost.

Natural Gas: The price of 32.0 cents per million Btu has been assumed. If 1070 is taken as the average Btu content per cu ft of gas, then the price per 100 cu ft becomes 3.42 cents.

Electric Power: This price is controversial because of differences of opinion by accountants in calculating the value of power produced from waste heat boiler steam. It will be taken here as 7.5 mils per kw-hr. Compressor power requirement will be assumed to be the round figure of 100 kw.



The Ajo reformer plant during construction.

The Ajo reformer plant—foreground, compressor; center background, catalyst chamber.

In the estimate of comparative costs, the statistics of gaseous refining, referring to a recent 30-day period of normal operation, are quoted below:

Anode Plant Statistics

No. of charges		58
Hr reduction time		172.75
Btu per cu ft natural gas		1070
Million Btu per ton anodes		0.204
Tons of copper refined as:		
	Anodes	8385
	Molds	63
	Launders	28
	Tap plates	3
	Residuum*	870
	Total	9349

* Because of the relative floor positions of furnace and spoon, the furnace cannot be completely drained at any time by casting anodes or other shapes. It has been estimated that a 15-ton residuum remains in the furnace after casting is terminated, necessitating subsequent re-oxidation and re-reduction with the succeeding charge.

From the above fundamental data, the following may be derived:

Total natural gas consumed	1.599 million cu ft
Natural gas/ton anode	191 cu ft
Natural gas/ton copper refined	171 cu ft
Gaseous Refining	**Anodes**
Production, tph	48.54
Hr per charge	2.98
Kw-hr/ton	2.060
Power cost, cents/ton	1.55
Cu ft natural gas/ton	191
Gas cost, cents/ton	6.53
Total cost, power + gas	8.08
Wood Poling	
Lb of poles/ton	43.0
Pole cost, cents/ton	64.5
Comparative Costs, Gas vs Poles	8.08/64 = 0.125

The economy resulting from the use of the air compressor during oxidation and skimming phases may be included in the comparative cost calculations, if so desired.

It is still too early in our experience with the new reformer and modified plant to have arrived at a realistic estimate of probable maintenance and repairs. To date, it does not appear to be an important item.

Miscellaneous comments

In the reduction of Cu_2O in molten copper with H_2 or CO, it may be determined by calculation and confirmed by actual plant observation that the heat of reaction is slightly exothermic. Processing a charge with reformed gas instead of poles produces no great difference in bath temperature. At times, the charge needs no extraneous heat and at other times heating with a burner is required to prepare a molten charge for casting after reduction is terminated.

In addition, there seems to be no discernable difference in appearance between poled and gaseous refined copper. Furthermore, laboratory study of two successive charges of copper, one having been poled and the other reduced with reformed gas, revealed no essential analytical difference except to the extent ordinarily expected in the laboratory analysis of duplicate samples.

Conditions around the furnace floor are now extremely quiet and orderly compared to former working conditions with poles. The violent action within the furnace and periodic explosions no longer prevail. The absence of poles, and of accompanying debris around the furnaces, has simplified the problems of housecleaning and housekeeping and has reduced fire hazards. No pole storage area is needed in the smelter area, and of course, the hazardous transfer of poles from railroad cars or trucks to the storage pile and retransfer to the anode floor have been eliminated, thus bringing to a close a dangerous, untidy, and highly undesirable smelter operation. To date, there have been no accidents of any kind within the smelter attributable to gaseous refining.

Much more study is required to bring to light and conquer the remaining problems of copper oxidation and reduction. It is certain that many improvements, mechanically and economically, will continue to appear in this field of metallurgy.

Conclusion

The outstanding features of gaseous reduction of oxygen-containing copper have been described and discussed. Compared to reduction with wood poles, reduction with reformed gas has been found by experience gained during the processing of over 100,000 tons of copper 1) to eliminate a hazardous and untidy smelter operation, 2) to simplify the practice of reduction and, 3) to culminate economically in a very substantial decrease in cost of copper refining.

DECARBURIZATION

BY OPEN-COIL ANNEALING

Decarburization-annealing of steel coils to make porcelain enameling sheet is described. Practices for open and tight winding of coils, the gases used for decarburizing, and the major problems are discussed.

by D. J. Blickwede

Special treatment of open wound coils of strip is an item in the long list of ideas conceived before the existence of a benign developmental climate. Thus, in 1903, Richard Varley[1] proposed to open wind a coil, and then fill the space between laps with a plastic material to be baked into the coil for electrical insulation.

Additional suggestions involving open winding were made over the years. Some had the same purpose as Varley's, but others had the purpose of obtaining faster heating and cooling during the anneal of a coil.[2] It was this feature that caught the attention of the steel industry a few years ago. However, we had other applications in mind. These included changing the chemistry of the steel by exposing the entire surface of a coil to special annealing gases.

For example, one application was suggested by our early laboratory tests on porcelain enameling of steel. These showed the carbon in steel to be a liability to optimum porcelain enameling properties, because it lowers transformation temperature, decreases the resistance to sagging, and frequently causes defects on the surface of the enameled article. Even enameling iron, with a carbon content as low as 0.015 pct, is not free of these troublesome effects. Unfortunately, steel cannot be refined to carbon contents lower than enameling iron, because the carbon-oxygen equilibrium of the steelmaking process prevents it. Therefore, removing the carbon from the strip in the annealing operation had great appeal as a way to make a superior steel for porcelain enameling.

Bethlehem has been producing very low-carbon sheet steel, made by open-coil decarburizing, since July, 1958; it has gained wide acceptance in the porcelain enameling industry. (US Patent No. 2,956,906, issued to Bethlehem, covers vitreous enameled articles made from this material.) Detailed description of the properties of the material is given elsewhere.[3, 4]

This paper is concerned with principles of the decarburizing process and open-coil-annealing practices. And it may be helpful to note how open-coil decarburization fits into the operation of making sheet steel. There are three steps to the process, conducted after tandem rolling and before skin rolling: specifically 1) open winding, 2) decarburize annealing, and 3) tight winding. The starting material is ordinarily rimmed steel.

Open winding

This is probably the easiest of the operations, a fact exemplified by the crude unit, shown in Fig. 1, that was used for our first experiments. It ran at only 100 fpm, which is too slow for production; so, we now use a manufactured unit which runs faster and has other advantages.

The purpose of open winding is, of course, to produce a uniform space between each lap of the coil. Laps that touch each other may not be in contact with the annealing gas. This is no problem, if the only concern is to heat and cool rapidly, but when the aim is to produce complete decarburization, edge to edge and end to end of a coil, contact between laps must be avoided.

One scheme for opening coils is to wind string between the laps, which is then pulled out, leaving a space between each lap. If the coil is not wound too tightly, this may be done quite readily with any string of proper diameter. The trouble with this

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method is that the laps do not stay put after the string is withdrawn. This is aggravated by moving the coil from the winding mandrel to standby storage and to the annealing pedestal.

We have successfully avoided this problem by using a specially designed, kinked, wire spacer. (Patent applied for. Made by Sobel Co., Easton, Pa.) Made from 0.018-in. high-carbon steel wire that has been bent in two directions (Fig. 2), it correctly spaces the laps of a coil regardless of how it is placed between them, does not snag when uncoiling from a spool, has enough strength to space heavy-gage strip, and allows free passage of gas between the laps of the coil. It is left in the coil throughout the anneal, and is not removed until the coil is finally tight wound. Although the spacer wire is discarded after annealing, the cost is nominal in view of these benefits:

1) It gives very uniform spacing, and prevents springback of the inner and outer laps;

2) The coil may be moved from place to place without the laps shifting;

3) Scratches that result from laps rubbing against each other are prevented;

4) The coil stays cylindrical during the anneal and does not warp out of shape; this is important in preventing coil-breaks later when tight winding; and

5) It permits open winding of heavy-gage coils (we have even handled 10-gage coils successfully); the same wire is used for coils of every gage.

The inside diameter of the open-wound coil has an important bearing on the coil-break problem in later tight winding. The usual 24-in. ID used in most mills is quite satisfactory for open-wound coils that are 20-gage and lighter. However, coil-breaks tend to occur in coils heavier than 20-gage when the inner laps are unwound after annealing. This is prevented by open winding heavy gage coils to large inside diameters, as follows:

20-gage and lighter—24-in. ID
16- through 19-gage—30-in. ID
15-gage and heavier—40-in. ID

Necessarily, open-wound coils are prone to edge damage, and must be handled with more care than tight-wound coils. A special magnet is used to lift opened coils.

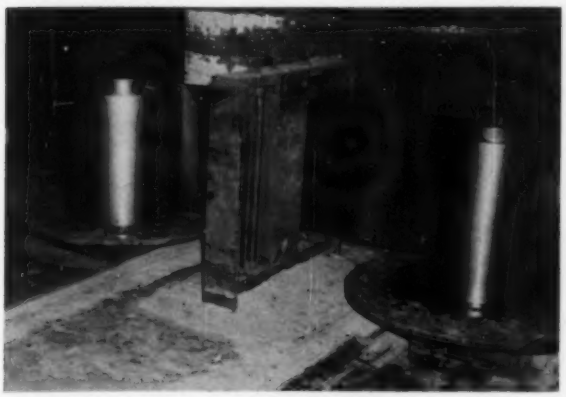

Fig. 1—Open winding unit used in first experiments. A new, manufactured unit is now used in production.

Decarburization anneal

The purpose of this step in the operation is to remove the strains of cold rolling by recrystallization, as well as to remove the carbon from the strip. Recrystallization starts and ends before decarburization takes place. As shown in Fig. 3, the grain size grows during subsequent decarburization at 1300°F, but not to the extent that drawability is harmed. It is fortunate that recrystallization takes place before decarburization; otherwise, the grain size would be too coarse.

Heating rates of opened coils are, of course, faster than those of tight coils. Typical comparisons have been published previously.[5]

Our annealing has been done on both 72-in. diam, three-pedestal bases, and 116-in. diam, single-pedestal bases. We have annealed one, two, and three coils per pedestal, with various gas flows. Despite these wide differences in conditions, the heating time is mostly dependent on the total weight of coils in the charge.

Decarburization Principles

There are three gases that can be used to decarburize steel: hydrogen, carbon dioxide, and water; viz:

$$\underline{C} + 2\,H_2 \rightleftharpoons CH_4 \qquad K_1 = \frac{pCH_4}{pH_2 \cdot a_c} \qquad [1]$$

$$\underline{C} + CO_2 \rightleftharpoons 2CO \qquad K_2 = \frac{pCO^2}{pCO_2 \cdot a_c} \qquad [2]$$

$$\underline{C} + H_2O \rightleftharpoons H_2 + CO \qquad K_3 = \frac{pH_2 \cdot pCO}{pH_2O \cdot a_c} \qquad [3]$$

($\underline{C}$ indicates carbon dissolved in steel.)

Equilibrium for each of these reactions is given by the equations at the right. In these, K_1, K_2, and K_3 are the equilibrium constants which depend on temperature, the subscript p's are the partial pressures of the gas constituents (vol pct divided by 100), and a_c is the activity of carbon dissolved in the steel. The last named is a measure of the tendency of the carbon to react with the gases; it depends on temperature and the carbon content of the steel. Values of K for these reactions and of a_c have been published.[6]

Whether or not a gas is decarburizing or carburizing is determined by comparison of the ratio of the products to reactants with the appropriate equilibrium constant. In practice it turns out that the first reaction—decarburizing with hydrogen—is very slow at annealing temperatures, and is thus unattractive.

Carbon dioxide decarburizes rapidly at annealing temperatures, but at lower temperatures tends to deposit soot. Fig. 4 shows this for a typical DX gas composition. Note that the ratio, $\frac{pCO^2}{pCO_2 \cdot a_c}$ of DX gas is less than the equilibrium constant at 1300°F (and so would decarburize steel), but it is greater below 1200°F (and so would tend to deposit soot). Of course, by using another gas for heating and cooling, and DX gas at the annealing temperature, the danger of sooting might be avoided. Nevertheless, we do not choose DX gases for decarburization treatments.

The best practical gas for decarburizing is water vapor, however, it will also oxidize steel. The reaction is:

$$Fe + H_2O \rightleftharpoons FeO + H_2 \qquad K_4 = \frac{pH_2}{pH_2O} \qquad [4]$$

The equilibrium constant, K_4 for this reaction at different temperatures has also been published.[5] It is shown as a function of temperature by the curve running slightly downward from left to right in Fig. 5. By the same rules mentioned earlier, the tendency of a gas containing hydrogen and water to oxidize the steel can be predicted by comparing the hydrogen:water ratio with the known equilibrium constant. Thus, gases with ratios above the equilibrium curve in Fig. 5 will not oxidize the steel, but those below will.

The decarburization tendency of non-oxidizing hydrogen-water gases can be predicted from equation [3], transposed as follows:

$$\frac{pH_2}{pH_2O} = K_3 \frac{a_c}{pCO}$$

When the proper values for K_3 and a_c are put into this equation, and various CO contents are assumed, the H_2:H_2O ratios in equilibrium with the carbon in the steel can be calculated. This was done (assuming a carbon content of 0.05 pct for a_c values) for CO contents of 0.5 pct and 10 pct, and the results are shown as the curves that go upward from left to right in Fig. 5. Ratios of H_2:H_2O below these curves will decarburize the steel, but ratios above will not. The CO contents of 10 pct and 0.5 pct were chosen because they correspond to the amounts at the beginning and end of a commercial decarburization cycle.

A range of temperatures and H_2:H_2O ratios can be defined at which decarburization will take place without oxidation. This is described in Fig. 5 by the area below the curves for decarburization equilibrium, and above the curve for oxidation equilibrium. It will be pointed out later that, in practice, there are also upper and lower temperature limits (because the decarburization rate is too slow at high and low temperatures). Thus, the practical range of temperatures and H_2:H_2O ratios is shown by shaded area in Fig. 5. Our practice is to work about in the middle of this range, i.e., at 1300°F and with H_2:H_2O ratios around 5.

One last point should be mentioned before leaving the thermodynamics of the process: It is the characteristic of H_2:H_2O gases to be oxidizing at low temperatures, but non-oxidizing at high temperatures. For example the gas with the H_2:H_2O ratio of 5, shown in Fig. 5, is above the equilibrium line at 1300°F, and is non-oxidizing. But it is below the line and will oxidize steel below 850°F. Therefore, the gas in which the steel is heated to, and cooled from, the decarburizing temperature must have a low H_2O content (and, hence a high H_2:H_2O ratio) to avoid oxidation. This gas will also decarburize, but very slowly; this is why the moisture content must be raised at high temperature for practical decarburization treatments.

Decarburization Rate

The preceding section about the thermodynamics of decarburization tells only whether or not reactions can take place. Whether they will in practice (or how fast they go) is another matter.

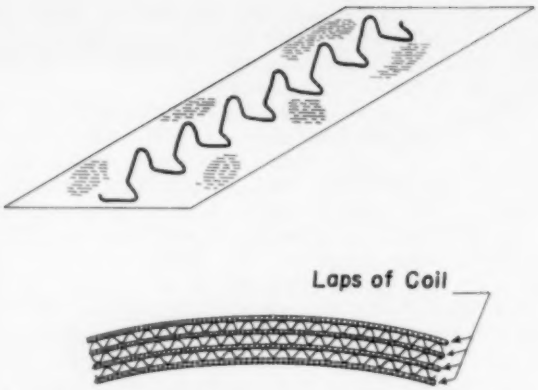

Fig. 2—Zig-zag wire spacer used to open-wind coils in preparation for decarburization; made from 0.018-in. diam, high-carbon wire.

The reaction rate depends on so many factors that it is difficult to predict. But it is useful to compare predictions with what actually happens, because this points out the factors that dominate and must be controlled for optimum results.

In the case of decarburization of sheet it can be imagined that one or another of these factors will affect the rate:

1) How fast the carbon diffuses through the steel to the surface, where it can react with the H_2O;
2) How fast the H_2O in the gas is brought to the surface of the steel to react with carbon;
3) How fast the reaction between C and H_2O at the surface takes place (this may be complicated by catalysis and side reactions); and
4) How fast the reaction products are swept away from the surface.

If the reaction is assumed to depend on the diffusion of carbon to the surface, the time it would take to decarburize 20-gage sheet from an original 0.08 pct C to a final 0.004 pct can be calculated from

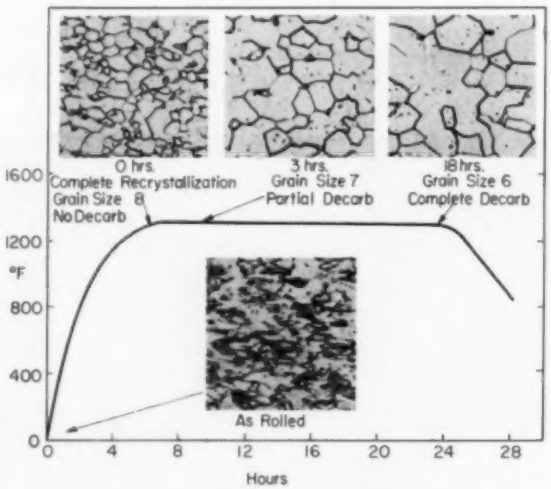

Fig. 3—Change in grain size during a typical decarburization anneal. Note that the steel recrystallizes before decarburization starts. 200X, nital etched.

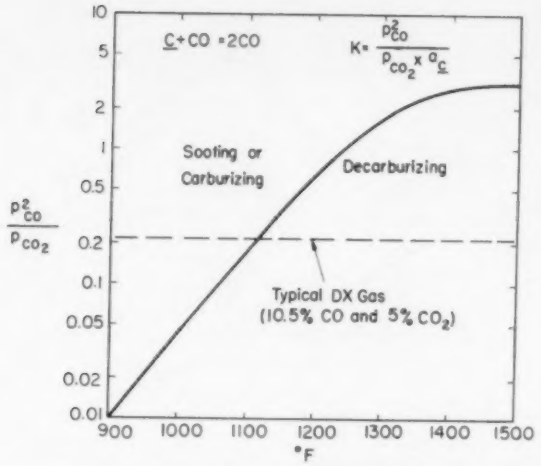

Fig. 4—Equilibrium between CO, CO_2, and 0.05 pct C steel. Note that DX gas will decarburize at high temperatures, but will deposit soot at low temperatures.

the known diffusion rates of carbon in steel.[7] Results of this calculation are shown by the curves in Fig. 6; several features are noteworthy:

First, because carbon diffuses faster in ferrite than in austenite, the inference is that the time for decarburization at 1300°F, where the steel is ferritic, is faster than at 1650°F, where it is mostly austenitic. At temperatures in between, different amounts of austenite and ferrite are present and a simple prediction of decarburization time cannot be made.

Superimposed on the curves in Fig. 6 are the ranges of times for decarburization that we have observed in laboratory tests (production results will be mentioned later); they correspond fairly well with the predicted times. It is particularly interesting to see that at temperatures much above 1600°F, decarburization slows down appreciably, because austenite is present. For this reason, and because the grain size becomes very coarse at high temperatures, our practice is to decarburize at or near 1300°F.

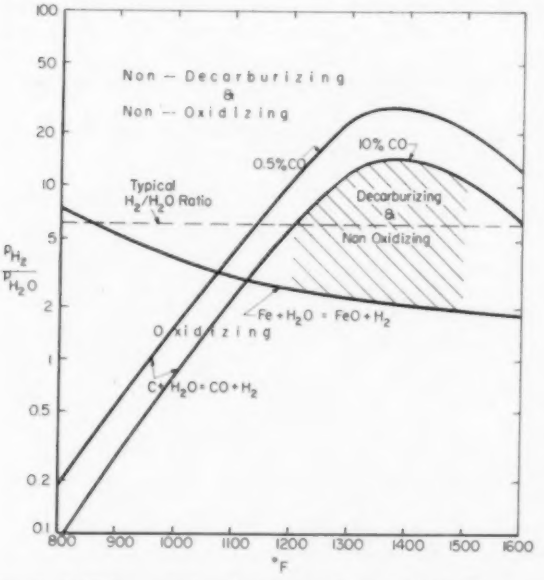

Fig. 5—Oxidizing and decarburizing tendencies of H_2:H_2O gases to 0.05 pct C steel.

The laboratory tests just mentioned were made with the deliberate attempt to keep at an optimum the other conditions that might control decarburization. In other words, ample H_2O was supplied for the reaction by using a small sample, and a large amount of wet, 18 pct H_2 gas (dewpoint was + 85°F, equal to about 3 pct H_2O, and 10 cu ft per hour was passed over a 0.01 lb sample). The high flow rate also tended to sweep away the CO as fast as it was formed. Under these conditions, decarburization rate is controlled by the diffusion of carbon to the surface of the steel. It will be seen later that, in production, conditions are not so ideal and other factors dominate.

Decarburization Practice

Naturally, our decarburization practice closely follows the principles just described; it could not work otherwise. This can be best illustrated by reference to Fig. 7. Plotted are the temperature of the control couple (which was 6 in. from the top and edge of the top coil), the temperature of the slowest heating part of the charge, and the gas composition under the cover. This is a typical heat on a large-diameter, single-pedestal base.

In our practice, two coils are heated to, and cooled from, the annealing temperature in a dry (−40°F dewpoint), 4 pct H_2, remainder N_2, gas. This is the standard H-N gas used in other places in our mill. This gas will not oxidize the steel and precludes the danger of explosion during heating.

When the control couple reaches 1200°F, the gas is switched to dry (−40°F dewpoint), 18 pct H_2 gas. This, too, is a standard gas in our mill, and is made either directly with an HNX gas generator, or by blending cracked ammonia with H-N gas. At this temperature and above, it may be used without danger of explosion.

Experience has shown that when the control couple reaches 1300°F, the parts of the coils that heat slowest are above the temperature at which oxidation will occur in moist 18 pct H_2 gas. Decarburization is then started by admitting steam under the annealing cover, along with the 18 pct H_2 gas. The amount of steam admitted varies from a great deal at the beginning of the cycle to very little at the end, and is controlled automatically by the dewpoint of the exit gas. Any dewpoint that will keep the H_2:H_2O ratio within the area shown in Fig. 5 is satisfactory: We generally stay in the middle, and control at about 70°F dewpoint.

As decarburization takes place, moisture in the atmosphere is consumed and hydrogen and carbon monoxide are generated. Also some carbon dioxide is formed by the following reaction:

$$H_2O + CO \rightleftharpoons CO_2 + H_2 \qquad [5]$$

Thus, as shown in Fig. 7, the hydrogen content of the atmosphere rose from 18 pct to about 22 pct, the CO content went from essentially 0 to 6 pct, and the CO_2 content went from 0 to 1 pct.

Probably the most noteworthy difference between the production results and the theoretical prediction is the time it takes to decarburize the steel. In the laboratory it took about 1 to 2 hr to decarburize 20-gage sheet, and this checked with the prediction based on diffusion rate of carbon. But in the production heat of 20-gage coils, shown in Fig. 7, decarburization took about 24 hr. Rate of the reaction in

production clearly is being controlled by factors other than diffusion of carbon in the steel. The few simple calculations which follow give a clue to these factors.

In the two coils of the illustration in Fig. 7 there was a total of about 41 lb of carbon (82,000 lb x 0.05 pct C). Now, if the reaction rate depended only on diffusion of the carbon out of the steel, it would take 2 hr to decarburize this charge, and carbon would have to be removed at the rate of about 20 lb per hr.

As shown by reaction [3], at least 18 lb of water is required for every 12 lb of carbon removed (assuming no side reactions between water and other constituents). Thus, for the case at hand, 30 lb of water per hr would have to be supplied to support a diffusion-controlled reaction. At standard temperature and pressure, this is 600 cu ft of H_2O per hr:

$$\frac{30 \text{ lb } H_2O}{\text{hr}} \times \frac{454 \text{ g}}{\text{lb}} \times \frac{1 \text{ mol}}{18 \text{ g}} \times \frac{22.4 \text{ l}}{\text{mol}} \times \frac{3.53 \times 10^{-5} \text{ ft}^3}{\text{liter}} = 600 \text{ ft}^3 \text{ } H_2O/\text{hr}.$$

If this was to be supplied by a moist HNX gas (say, 18 pct H_2, 3 pct H_2O) a minimum of 20,000 cu ft of the HNX gas per hr would be needed. This is an impractical amount to handle, and also would be very costly. It is easier to inject steam directly into the annealing cover, along with the dry 18 pct H_2 gas, than it is to use pre-humidified 18 pct H_2. However, steam injection still falls short of accomplishing the reaction in theoretical time.

At the start of the cycle, the carbon content at the surface of the sheet is high and it quickly reacts with the water in the atmosphere. As decarburization progresses, the carbon content of the surface of the steel drops; so, it must diffuse longer and longer distances from the interior of the sheet to reach the surface. Thus, the reaction slows down, and water is not used up so fast. For this reason, the amount of steam injected must be constantly reduced during the cycle; otherwise the moisture content of the atmosphere would become high enough to oxidize the steel. The best way we have found to do this is to control the amount of steam injected to maintain a constant dewpoint of the exit gas. In this way we provide all the reaction demands, without running the risk of supplying so much that the steel would oxidize.

The above suggests that the decarburization time in production would depend on the gage of the steel, the weight of the steel charge, and the carbon content. As shown in Fig. 8, actual decarburization time depends mainly on weight charged.

Problems in Decarburization

Needless to say, there are many problems; careful attention to the process is needed to assure consistent success. Here are a few that have come up, but are now under control:

1) Shape of the coil: Poor shapes can result from annealing a coil that was open wound with string on a pedestal that was not flat. Winding the coil with the permanent wire spacer and machining the pedestal flat prevents such warping.

2) Oxidation or scaling during the anneal: If moisture is introduced under the annealing cover before the slow heating portions of the charge are at a high enough temperature, oxidation of the surface may result. Unfortunately when this happens, the oxide layer is not completely reduced during subsequent decarburization.

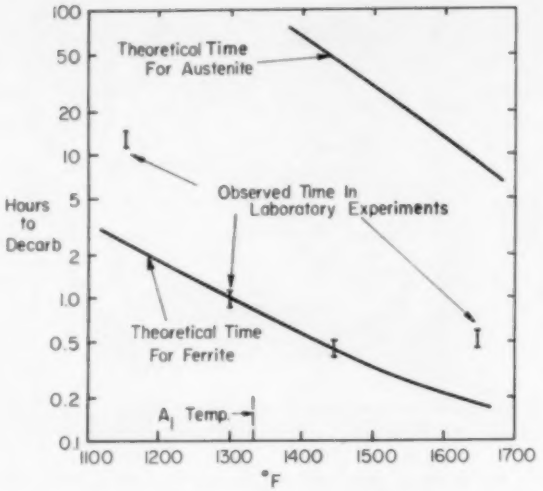

Fig. 6—Time to decarburize a 20-gage sheet (0.036 in.) from 0.08 to 0.004 pct C.

3) Incomplete decarburization: The effectiveness of the decarburization treatment can be determined by a metallographic examination for carbides in the structure. Cross checks with chemical analyses have shown that, if no carbides are present after a picral etch and micro-examination at 1000X, the carbon content of the steel is below 0.01 pct, and is probably below 0.003 pct. On this basis, we currently have less than 1 pct rejects for incomplete decarburization.

However, this record was not obtained without solving some difficulties in the initial experiments. For example, we found that coils open wound with string were prone to incomplete decarburization;

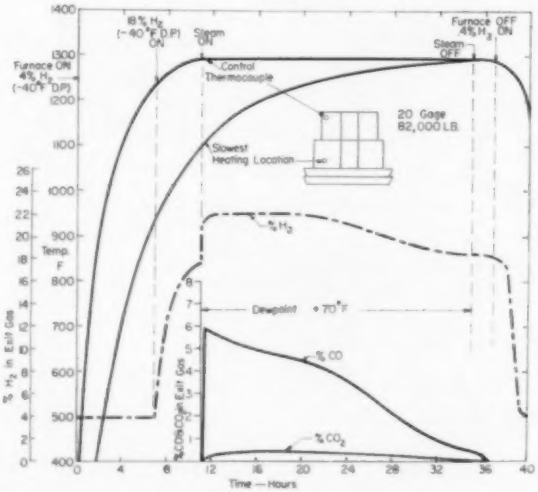

Fig. 7—Typical coil temperature and gas composition during decarburization of open-wound coils

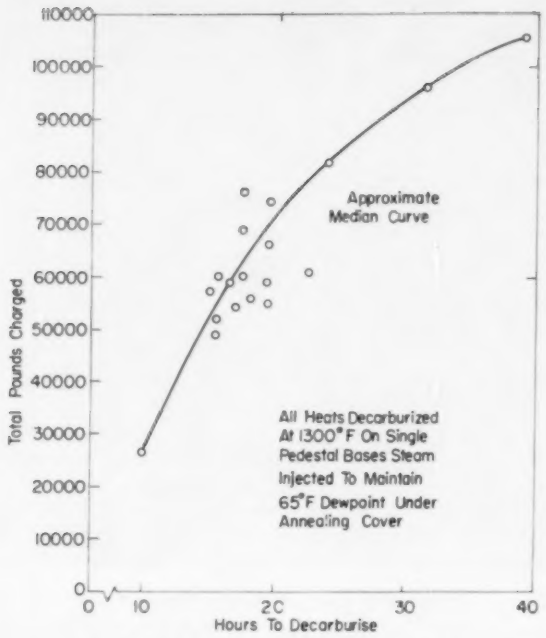
Total Pounds Charged
Hours To Decarburise
Approximate Median Curve
All Heats Decarburized At 1300°F On Single Pedestal Bases Steam Injected To Maintain 65°F Dewpoint Under Annealing Cover

Fig. 8—Decarburization time vs coil weight.

the wire spacer solved this. Also, as mentioned above, oxidation during the heating stage sometimes caused incomplete decarburization; this was solved by proper control of the gas under the cover during heating.

Internal Oxidation

This should actually be listed with the above as the fourth problem in decarburization. However, internal oxidation has such an important effect on properties that it is a subject in itself. Little is known about it; so, we have been conducting some careful investigations in the laboratory, results of which we hope to publish in detail at a later date. For the present purpose, only a brief resumé of the phenomenon will be given, and emphasis will be placed on the prevention rather than the cause.

It is well known that certain alloys are prone to internal oxidation if heated in atmospheres that do not form an oxide on the surface.[8,9] Internal oxidation is caused by the diffusion of oxygen into these alloys, where it combines with the alloy elements that have a high affinity for it. For example, the phenomenon occurs in copper-beryllium, iron-silicon, iron-chromium and iron-aluminum alloys.

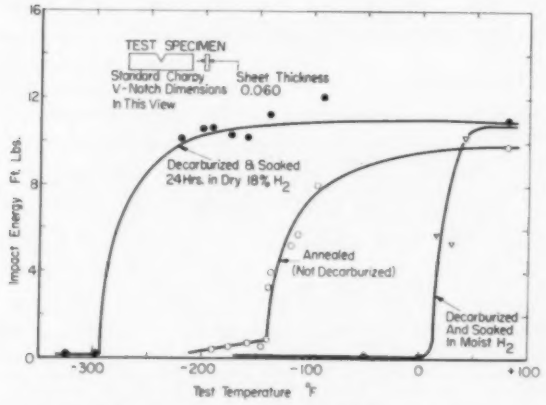
TEST SPECIMEN
Standard Charpy V-Notch Dimensions In This View
Sheet Thickness 0.060
Decarburized & Soaked 24 Hrs. in Dry 18% H2
Annealed (Not Decarburized)
Decarburized And Soaked In Moist H2
Impact Energy Ft. Lbs.
Test Temperature °F

Fig. 9—Notch-bar impact curves for sheet. Note that the temperature at which the steel became brittle was raised by internal oxidation, but was markedly lowered (improved) by subsequent treatment by dry 18 pct H_2.

We have also observed internal oxidation in decarburized rimmed steel which appeared as a zone of fine precipitates near the surface of the steel. Electron diffraction showed the precipitate to have the crystal structure of Fe_3O_4; yet we also found that it did not occur in pure, zone-refined iron. Perhaps it was an alloy oxide of the type $(Fe, M)_3O_4$ (where M signifies the alloy or residual element).

Not all rimmed steel, that has been decarburized, contains internal oxidation. Laboratory experiments show that it forms only *after* the steel has been decarburized, during prolonged holding in moist atmospheres. Production heats like that described previously in Fig. 7, with which the moist hydrogen atmosphere was discontinued as soon as decarburization was complete, do not contain internal oxidation. In other words, by proper control of the anneal, internal oxidation can be prevented. Also it will be shown later that, even if internal oxidation should occur, the harmful effects can be cured.

The precipitate formed during internal oxidation may be distributed in two ways: within the grains and along grain boundaries. In the initial stages of formation, very fine particles appear in a narrow band near the surface of the steel. At advanced stages, the precipitate tends to be somewhat coarser and partially distributed along the grain boundaries. The rate of internal oxide formation is proportional to the temperature and moisture content of the gas.

Concurrent with the progress of internal oxidation after decarburization, the notch ductility of the steel becomes poorer. There is no detachable change in the room temperature tensile or Olsen properties, but the ductility becomes nil at very low temperatures. Thus, Fig. 9 shows that the ductile-brittle transition temperature of decarburized sheet that had a prolonged soak in moist hydrogen gas is much higher than the same material that was not soaked after decarburization. The fracture of the embrittled specimen followed the grain boundaries, whereas the fracture of the ductile specimen was transgranular.

Fig. 9 also shows that the ductility of the embrittled steel can be restored by a prolonged treatment in *dry* 18 pct H_2 gas. In combination with the other facts concerning this phenomenon (the concurrently formed internally oxidized zone near the surface and the intergranular fracture), this suggests very strongly that the embrittlement is due to oxygen at the grain boundaries. Evidently, if there is carbon at the surface, oxygen does not diffuse into the steel, either to locate at the grain boundaries or to combine with alloy or residual elements. But holding in the decarburizing gas after the carbon has been removed does cause the steel to take up oxygen. Oxygen seems to be taken up in two forms: as internal oxide particles in a band near the surface, and dissolved at the grain boundaries.

Embrittlement of steel after treatments in wet hydrogen has been observed before,[10, 11, 12] but there

has been some controversy on whether it is due to dissolved oxygen, or to the mere absence of carbon. Our work shows that dissolved oxygen is the culprit.

With proper control, very low carbon steel can be made with excellent ductility; indeed, we are doing it all the time. Experience has shown us that on single pedestal bases, with the practices described, discontinuing the decarburization treatment when the atmosphere reaches 0.4 pct CO avoids the dangers of internal oxidation and embrittlement. However, on three-pedestal bases it sometimes happens that the coils on one pedestal will be completely decarburized before the coils on the others. In this event, if it is not possible to change the gas on the pedestal to dry hydrogen, internal oxidation may occur. However, the harmful effect on ductility can be corrected by soaking the coils on all pedestals in dry hydrogen as soon as the last one has finished decarburization. Tests show that the higher the hydrogen content of the dry gas, the less the time required for restoring ductility.

Tight winding

With decarburization completed, the open coil must now be re-tight-wound so that it can be handled in skin rolling, the next operation. The principal problem here is to prevent scratches and coil-breaks. It was mentioned earlier that scratches are prevented by taking the necessary precautions to assure cylindrical coils after the anneal. Also, the wire spacer is of great benefit.

Coil-breaks are prevented by using special equipment. In essence, this equipment unwinds the annealed open coil around guide rolls, which support the strip in a vertical position; and then it passes the strip between a snubber roll and a 5-ft diam rubber-covered tracking roll, and finally onto the wind-up mandrel. The 5-ft rubber roll has regenerative braking so that proper back tension can be applied to the strip as it is wound on the mandrel.

In the annealed condition, decarburized sheet has less tendency to flute and form coil-breaks than the rimmed steel from which it was made. However, coil-breaks can still occur. There are two main sources: one is the unwinding of the inner laps of the annealed open coils, and the other is in tight winding the inner laps of the tight coil. The problem is greater with heavy-gage, wide strip.

We handle the problem by open and tight winding heavy gage strip on large inside diameters. Our practice for open coils, was described earlier; all our tight coils are wound to 30-in. ID. They require a special adapter to be handled on the skin mill mandrels. During tight winding the wire spacer is removed from the coil, wound on a spool, and discarded.

Discussion

It was mentioned at the beginning that open-coil-annealing may be used to accomplish changes in the chemistry of sheet steel that cannot be done in steelmaking. Emphasis so far has been on decarburization, but mention should also be made of denitriding.

It is well known that moist, pure hydrogen may be used to denitride steel. The reaction is thought to be:

$$2\,N + 3H_2 \rightleftharpoons 2\,NH_3 \qquad [6]$$

Moisture is believed to act as a catalyst for the reaction.

When nitrogen and carbon of rimmed steel are both reduced to very low amounts, the steel becomes non-strain aging. Thus, it would be desirable to denitride as well as to decarburize the steel. Unfortunately, denitrification by the above reaction takes place much more slowly than decarburization, and it is even slower in hydrogen-nitrogen gases. For this reason, strain aging is not eliminated by the decarburization practice that has been described.

Summary

Essential details of open-coil decarburization have been described in the foregoing paragraphs; the following are highlights:

1) Open wind the tandem reduced coil with a wire spacer. Heavy-gage coils are wound to larger inside diameters than those of light-gage.

2) Coils are double-stacked on either single or multiple pedestal bases and heated to 1200°F in 4 pct H-N gas with a dewpoint of −40°F.

a) When the control couple reaches 1200°F, 18 pct H-N gas with a dewpoint of −40°F is admitted.
b) When the control couple reaches 1300°F, steam is admitted along with the dry 18 pct H_2 gas. The amount introduced is continuously controlled throughout decarburization to maintain a dewpoint of +70°F. Coils are maintained at about 1300°F throughout decarburization.
c) When the CO content of the gas under the cover drops to 0.4 pct the steam is turned off, and the coils are purged 1 hr in dry, 18 pct H_2 gas. Temperature is maintained at 1300°F.
d) On three-pedestal bases, coils are given an additional purge of 10 to 24 hr to overcome the embrittling effect of internal oxidation.
e) After the purging period, dry, 4 pct H_2 gas is admitted, and the coils are cooled.

3) The decarburized coils are tight wound, and the wire removed and discarded. Heavy-gage coils must be tight wound on larger inside diameters than light-gage coils to prevent coil-breaks.

4) The decarburized rimmed steel coils have excellent properties for porcelain enameling.

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Vitro's Salt Lake plant

SCANDIUM RECOVERY FROM URANIUM SOLUTIONS

The commercial recovery of scandium as a by-product of uranium extraction is described. A flouride strip system was developed to recover scandium from the solvent in concentrated form, and high purity scandium oxide was prepared in multi-pound lots by chemical separation techniques.

by L. D. Lash and J. R. Ross

Scandium is a pseudo-rare earth which is truly rare and expensive. It has special properties which may make it desirable even at the present price of $2750 per lb. Recently the price was lowered from $5000 per lb which had prevailed since 1952. It is anticipated that usage will be stimulated because of the lowered price.

This metallic element is distributed widely in trace amounts in the rocks of the earth's crust. In addition to micro-amounts found in most uranium ores, concentrations of 50 to 100 ppm have been found in Colorado ferberite ore, lateritic nickel ore, heavy sandstone from Utah, Wyoming, and New Mexico, and various zircon sands and monazites from the western part of the US. Scandium has been found as an essential constituent of very few minerals, the most important being thortveitite, a scandium silicate. At the present time, this mineral commands a price more than twice that of gold.

Chemically, scandium is in group 3A of the periodic table. It is closely associated with yttrium and the rare earths which it strongly resembles in its reactions. It has a valence of three and will form an insoluble hydroxide, fluoride, or oxalate under proper conditions, similar to the rare earths. Eight years before its discovery, scandium was predicted by Mendeleef who ascribed his *eka-boron* with properties nearly identical to the actual properties of scandium. Its position as the element with Atomic No. 21 and Atomic Wt 44.96 places it between calcium and titanium in Period 4 of the chart.

The successful commercial application of scandium must depend on unique properties to justify use of this high cost substance. Greater consumption would allow additional price reductions, but scandium will remain a relatively high-priced commodity because of its inherent scarcity. Research reportedly has been oriented toward use of scandium as an agent to achieve high-temperature, low-density alloys with magnesium and tantalum.[1] Also, the US Air Force sponsored a project for production of research amounts of pure scandium metal.[2] The oxide has been used experimentally in electronics, ceramics, and metallurgy.

Excellent reviews of the subject of scandium are given in *Mineral Facts and Problems*[3] and in a Report of Investigation to be published by the Salt Lake City Metallurgy Research Center of the Federal Bureau of Mines. A. H. Daane of the Ames Laboratory presented new information on scandium metal

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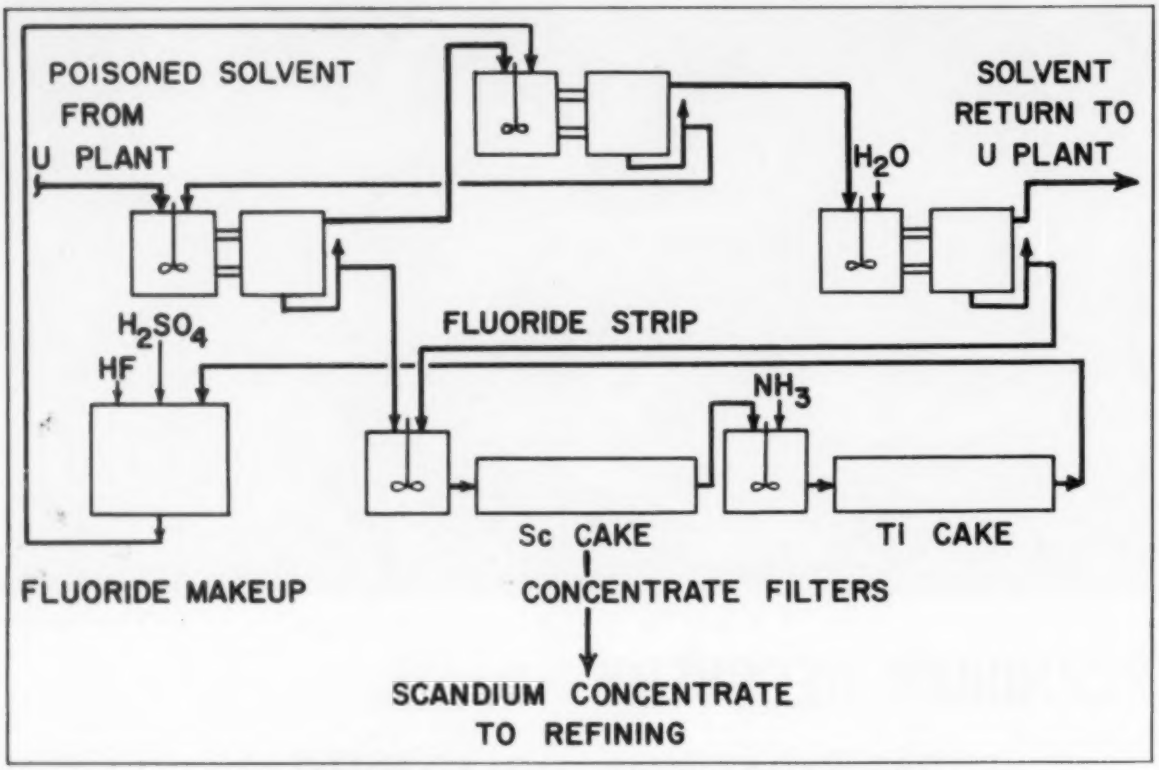


Salt Lake plant flow sheet showing the use of hydrofluoric acid strip for scandium recovery from uranium plant solvent.

at the recent conference on *Rare Earth Research Developments* at Lake Arrowhead, Calif.[4] In addition, a recent book by R. C. Vickery summarizes information on the chemical behavior of scandium and its compounds.[5]

Concentrate recovery

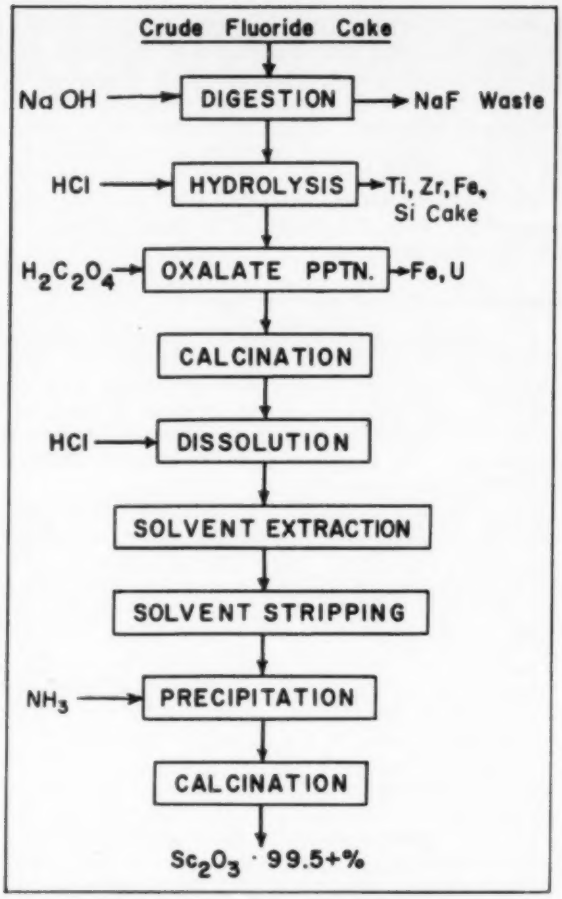
Small amounts of scandium exist in uranium ores and are dissolved during an acid leach yielding up to 0.001 g per liter Sc_2O_3. When Vitro converted their uranium plant from phosphate precipitation to solvent extraction,[6] scandium was found to follow the uranium into the dodecyl phosphoric acid solvent (DDPA). However, scandium did not strip with uranium from the DDPA in hydrochloric acid, but remained in the solvent. Therefore, a concentration of scandium built up in the organic phase. Residues from the solvent were spectrographed by the Bureau of Mines and found to contain scandium.

As a result of this discovery, provisions were made in the plant for recovery of scandium-bearing concentrates. Solvent extraction of uranium was added to a conventional acid leach process in a typical installation.[7] The ore was crushed and ground, and then leached with dilute sulfuric acid. Addition of an oxidant such as sodium chlorate insured conversion of uranium minerals to a soluble form. At this point, the slurry was chemically reduced by a sulfide, such as sodium hydrosulfide, to remove substances such as ferric iron and molybdenum which were partially extracted by the solvent. The solids-liquid separation was accomplished by a four-stage CCD thickener circuit. The uranium was extracted from pregnant liquor with 0.1M DDPA in kerosene and stripped with 10N HCl. The stripped solvent was recycled to the extraction section and used again. Most of the hydrochloric acid was evaporated and recovered for re-use. Following ammonia precipitation and filtration of yellow cake, the uranium was calcined to an oxide product which was shipped to the Atomic Energy Commission.

However, the hydrochloric acid uranium stripping agent did not strip scandium, thorium, or titanium from the DDPA solvent. These three elements continued to selectively extract into the organic phase at the expense of uranium loading. The solvent became unable to properly extract uranium, because the exchange sites were progressively filled with non-strippable metals. This effect is known as poisoning of the solvent.

A hydrofluoric acid stripping section was incorporated in the plant to remove thorium and titanium poisons from the solvent. It was later found that the hydrofluoric acid circuit also removed scandium; therefore, a study was initiated to optimize recovery of scandium in this section. Laboratory studies with continuous mixer-settlers were used to investigate the fluoride stripping variables, such as flow rates, fluoride concentrations, and phase separation rates. These data were scaled up to design size for the mill operation.

For scandium concentrate recovery, a portion of the plant-stripped solvent was fed to the hydrofluoric strip section for removal of the poisons. Typical scandium content was less than 0.1 g per liter Sc_2O_3 in the poisoned organic. The solvent was contacted in a two-stage, countercurrent system with an acid fluoride solution to remove scandium and thorium as precipitates and titanium in soluble form. Organic feed rate was 10 gpm, or about one-tenth of the uranium plant organic stream. Aqueous fluoride feed rate was 1 gpm, resulting in a 10:1 organic to aqueous ratio. The fluoride solution contained 6 pct fluoride and was adjusted to a pH of 4



Thorium-Scandium cake is refined at the Chattanooga, Tenn. plant to high-purity scandium oxide by chemical and solvent extraction methods.

with sulfuric acid. Mixer-settlers with adjustable gravity overflows were used for contactors in these solvent stripping stages. This allowed removal of the fluoride precipitates formed during stripping. After washing with a small amount of water in an additional single mixer-settler stage, the solvent, with poisons removed, was recycled to the uranium extraction section.

The main aqueous stream was filtered to remove the scandium-thorium fluoride cake, yielding a product containing approximately 10 pct Sc_2O_3 and 20 pct ThO_2. This concentrate was packaged in drums ready for shipment to the refining facilities. The filtrate was neutralized with ammonia to precipitate the titanium plus a small remaining amount of scandium and thorium. The solids were removed by filtration and stored for possible future recovery. The mother liquor was regenerated with hydrofluoric and sulfuric acids and recycled back to the strip section for further scandium recovery.

Equipment in the strip circuit had the difficult duty of resisting corrosive fluoride solutions and kerosene simultaneously. Saran-lined pipe was used with excellent results for transfer lines. The level-control legs on the mixer-settlers utilized saran tubing for adjustability. This was much less satisfactory than pipe and had to be replaced periodically. The flexible tubing was almost necessary, however, to handle the slurry formed by precipitation of fluorides in the stripping operation.

Neoprene lining was used in the tanks with fair success, although some deterioration had taken place in less than a year. Neoprene coverings were also used for the plate-and-frame presses.

In general, centrifugal pumps were used to transfer liquids and to feed the presses. Durimet-20 pumps performed well in this installation

Radiation hazards

The thorium-scandium cake was shipped to the Chattanooga, Tenn. plant of Vitro Chemical Co. for refining to extremely high purity scandium oxide. This concentrate exhibited a relatively large amount of radioactivity due to the thorium-230 present in the thorium fraction. As a result, it was necessary to enforce rigid rules of cleanliness and exposure around the filter presses which processed this concentrate. The exposed precipitate, especially immediately after drying, emitted strong beta radiation. This apparently resulted from the isotope thorium-234 which chemically followed the natural thorium. It had a 24½-day half-life and consequently dissipated fairly rapidly with passage of time. Also, strong alpha radiation was exhibited by thorium-230, a product in the uranium radioactive decay chain. It has a much longer half-life (80,000 years), approaching that of radium, and consequently continues alpha emissions with very little attenuation. This presented a lasting hazard when the precipitate was exposed. The alpha-rays were stopped by metal containers such as the 55-gal drums used for shipping. As a result, no difficulty was experienced in handling the concentrate after it was packaged.

In addition to scandium, it will be noted that this concentrate could provide a source of radioactive thorium isotopes such as thorium-230 (ionium)[8] and thorium-234.

Oxide purification

The thorium-scandium cake containing about 10 pct Sc_2O_3 was refined to high-purity scandium oxide at Chattanooga. The crude fluoride cake was digested in a 15 pct solution of sodium hydroxide for conversion to scandium hydroxide. Metathesis required 4 hr at temperatures between 75° and 90°C. Slurry was filtered in a rubber-covered plate-and-frame-press to remove soluble sodium fluoride and other impurities. The crude hydroxide cake from the filtration was digested in hydrochloric acid, and the pH adjusted to four for removal of contaminants by hydrolysis. This was accomplished by heating to 100°C to precipitate titanium, zirconium, iron, and silica. If large amounts of these elements were present, then the hydrolysis cake held considerable scandium and had to be recycled. The filtrate contained scandium, some iron, and uranium. Scandium was recovered from the solution by precipitation with oxalic acid under closely controlled conditions. For complete recovery it was necessary to use precisely the stoichiometric quantity of oxalic acid with no excess.

If large amounts of uranium were present, the solubility of scandium oxalate was dramatically increased. Although initial concentrates from Salt Lake contained enough uranium to color the thorium-scandium cake green with UF_4, later concentrates were almost free of uranium and presented no difficulty.

The oxalate cake was filtered to remove iron and uranium, then calcined at a relatively low temperature. It was imperative to maintain the roasting temperature at less than 800°C to avoid formation of a difficulty-soluble oxide.

Following solution of the scandium oxide in hydrochloric acid, a solvent extraction step was used to extract and further purify the scandium chloride solution. The extract was stripped to yield a high-purity scandium solution and ammonia was used to recover the hydroxide. Again, close control was required for complete recovery of scandium. The hydroxide was calcined at 700°C to produce scandium oxide with a purity greater than 99.5 pct Sc_2O_3. Further refining has produced products assaying 99.99 pct Sc_2O_3.

Analytical methods

Conventional chemical determinations are used for analysis of scandium in the intermediate ranges, but spectrographic methods are required by extremely high or extremely low concentrations. In the range of 0.01 to 50 pct Sc_2O_3 or more, the chemical method may be used. This involves an ether-thiocyanate extraction for removal of thorium[9] followed by an EDTA titration for determination of scandium.[10] The chemical separations schemes are varied depending on the specific samples. For instance, solvent samples are evaporated to dryness and the carbonaceous materials then taken into solution in oxidizing acids. Alternately, the fluoride samples which are not soluble in acids are converted to hydroxides by caustic metathesis and hydrochloric acid is used to dissolve the sample. Except for sample preparation, the complete chemical analytical methods are presented in the references cited.[9,10]

A low range spectrographic analytical method was developed at the Salt Lake Metallurgy Research Laboratory of the Bureau of Mines.[11] The method is reproducible and covers all ranges of scandium concentration with a standard deviation of approximately 10 pct. This deviation is insignificant at low concentrations, but develops into a sizeable problem of accuracy in scandium concentrates.

After solubilizing the scandium, an ammonia precipitate is made to concentrate the scandium before dehydration with perchloric acid to eliminate silica. A sodium peroxide separation eliminates aluminum, and a cupferron-chloroform extraction is made to remove iron, titanium, zirconium, vanadium, and similar elements. A solution containing 50 mg ZrO_2 and 0.25 mg TiO_2 is added before precipitating with ammonia hydroxide to form a common refractory zirconium matrix and to provide titanium as an internal standard.

An arc-spectrographic method for the determination of trace impurities in high-purity scandium oxide was developed by Vitro Chemical Co.[12] Standard samples were prepared by adding measured amounts of impurities to an ion exchange purified scandium oxide. A series of six standards covering an impurity concentration range of 50 to 1000 ppm of the following transition, alkaline earth and rare earth elements was prepared: Fe, Mn, Ti, Si, Ca, Mg, Zr, Th, U, Eu, La, Ce, Sm, Nd, Gd, Yb, and Y.

Samples to be analyzed along with appropriate standards are mixed with two parts of pure carbon and burned to completion in a 12-amp dc arc. A Stallwood Jet was used to suppress the CN band structures that are often detrimental to spectrographic analysis. The transmission readings obtained from the microphotometer were evaluated in the usual manner with a Respectra Calculator. Using the above elements, appropriate line pairs using scandium as an internal standard were selected as being most suitable for the evaluation of the spectra.

The results produced by this method are generally satisfactory with the standard deviation placed at less than 4.0 pct.

Conclusions and new advances

1) *Scandium is available*—An adequate supply of high purity scandium oxide is available for any forseeable use, and sizable inventories exist. Relatively large quantities of scandium occur in the leach liquors of uranium mills and are potentially recoverable. Also, the uranium mills are possible sources of ionium (thorium-230);

2) *Analytical methods*—New spectrographic techniques have been developed for high purity scandium compounds, where the impurities are assayed, and for low grade materials, where the scandium emission lines are used in the determination. Conventional chemical methods are available for assay of scandium in intermediate ranges of concentration;

3) *Scandium is expensive*—The high intrinsic price of scandium dictated by its scarcity and costly purification, limits its use to extremely specialized applications. Thus, it would normally be utilized in areas where only scandium, because of its unique properties, can be used. Through research now in progress, expanded markets could undoubtedly develop. Obviously, a larger sales volume will result in a much lower price; and

4) *New processes*—The Bureau of Mines, Salt Lake City Metallurgy Research Center, developed a scandium recovery process using solvent extraction with DDPA. However, they eliminated the necessity of chemical reduction of ferric iron, an expensive step, formerly necessary in scandium recovery as practiced in the uranium plant operation. Other solvents for scandium have also been tried. Recently a 5-gpm pilot plant has been operated on a cooperative basis, between the Bureau of Mines and Vitro. The portable pilot plant, which is mounted on a truck for versatility, embodies a complete prototype solvent extraction plant. Reviews of pilot-plant studies and other information are contained in the new Bureau of Mines Report of Investigations *Reconnaissance of Sources and Recovery Methods of Scandium.*

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HISTORY OF THE COKING INDUSTRY IN THE UNITED STATES

Part V

In this last article of a series, the coke industry of today is discussed and some interesting statistics concerning the industry are presented.

by C. S. Finney and John Mitchell

On December 31, 1959, there existed in the United States 15,993 slot-type coke ovens capable of producing 81,447,700 net tons of coke. These ovens were concentrated in 74 coke plants in 21 different states. As of the same date, there were 7448 beehive ovens in existence at 45 plants in the states of Pennsylvania, Virginia, West Virginia, and Kentucky. Total annual capacity of the existing beehive ovens was 4,368,800 net tons, but only 5148 ovens with a capacity of 3,131,600 tons were in operating condition.

It is interesting to compare the average dimensions of slot-type ovens built during recent years with the 30 ft x 5½ ft x 16½ in. ovens erected at Syracuse, N. Y. in 1892. A composite oven built according to the average dimensions of all those erected between 1954 and 1958, for instance, would be 39 ft long, 12 ft high, and 18 in. in width. The coal capacity would be 16 tons as against the 4.4 tons which could be charged to the Syracuse ovens. Of the 15,993 slot-type ovens in existence at the end of 1959, by far the greater number were built by the Koppers Co. whose total of 11,280 ovens included 7891 Koppers-Becker and 3389 Koppers ovens. Of the remainder, there were 3260 Wilputte, 1350 Semet-Solvay, 63 Otto, and 40 Simon Carves ovens.

By-product coke oven plants are usually classified either as *furnace* or *merchant* plants. According to the definitions used by the US Bureau of Mines, the former are "those that are owned by or financially affiliated with iron and steel companies whose main business is producing coke for use in their own blast furnaces. All other coke plants are classified as merchant. They include those that manufacture metallurgical, industrial, and residential heating grades of coke for sale on the open market; coke plants associated with chemical companies or gas utilities; and those affiliated with local iron works, where only a small part (less than 50 pct of their output) is used in affiliated blast furnaces." The annual coke capacity of the merchant plants during 1959 was 10,393,000 tons. However, the by-product oven of today is essentially an appurtenance of the iron and steel industry, rather more than 87 pct of total by-product coking capacity being concentrated at furnace plants.

This was not always so. There was a time when the merchant plants played a much greater part in meeting the US demand for coke and gas. High noon for the merchant plants was reached during the early 1930's. By 1932 there were as many by-product oven installations being operated by the merchant sector of the industry as by the coke divisions of the iron and steel industry (44 of each), and in the same year the merchant plants produced 46.5 pct of all by-product coke made in the country. Since that time their contribution has drastically declined. In 1940 merchant plants were responsible for only 23.2 pct of total US production, and by 1950 their number had decreased to 30 plants which turned out 18.5 pct of the total by-product coke made. At the end of 1959 only 20 of the 74 existing by-product oven installations were merchant plants. They accounted for 12.5 pct of the year's production, or 6,849,786 net tons. This percentage has remained fairly constant since 1954.

There are several reasons for the decline of the merchant coking industry. For example, on the grounds of economy, quality control, continuity of supply, and so on, the iron and steel industry usually prefers to control its own mines and carbonize its own coal at or near to the blast furnace rather than rely on independent operators for metallurgical coke. As the steel companies have enlarged their own coking facilities, so has the need for coke obtained from other sources declined. Furthermore, not only has the steel industry increased in self-sufficiency by building more coke ovens during recent years, but it has also progressively improved the fuel efficiency of its blast furnaces. During the years 1947-49 the average coke consumption per ton of pig iron was 1892.8 lb. During 1958 the corresponding figure was 1613.4 lb. There are many individual furnaces where still better results are being obtained, and further reductions in the average may be expected. Perhaps the greatest threat to the merchant coking plant has been the fantastic increase in the use of natural gas and petroleum products for purposes which manufactured gas once served. So deadly has the competition from natural gas and oil been that it has almost eliminated by-product oven installations owned by public utilities. In the peak years of the early 1930's there were 23 such public utility plants. In 1960 only two were left. One of these, owned by the Citizens Gas and Coke Utility, was at Indianapolis, Ind.; the other was the plant operated by the Philadelphia Electric Co. at Chester, Pa.

The non-utility merchant plants have also been sorely hit. With gas sales revenues reduced, domestic

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coke market almost gone, and sales of water-gas coke nearly eliminated by natural gas and oil, their difficult situation has been mitigated to a considerable extent by the foundry-coke market which in 1958 accounted for almost half of their total coke revenue, and by the elimination of competition from utility plants. The increasing availability of, and the widespread predilection for, natural gas, together with the consequent effects upon the production of manufactured gas are shown by the following data.

When on April 28, 1960, the very first of the coke oven plants built specifically to supply city gas ceased to operate at Everett, Mass. there must have been many who felt that the end of an era had been reached. The *Otto coke* which had competed with anthracite for grandmother's favor did not survive the more sophisticated demands of her daughters, and long since followed anthracite into the limbo. Today, the housewife of the greater Boston area may warm her home, cook her food, and heat her water with natural gas from fields far to the south.

At the end of 1959, of the 7448 beehive ovens in the United States, only 5148 were in operating condition. These could have produced 3,131,600 tons of coke, but actual production was only 1,074,296 tons. However, one mechanized beehive coke plant was placed in operation in 1959, and plans to build two other plants were reported.

1959 Production of Beehive Coke by States—net tons

Pennsylvania	713,150
Virginia W. Virginia and Kentucky	361,146
Total	1,074,296

The stronghold, if such it can be termed, of beehive coking is still the state of Pennsylvania, in which there remain 4125 of the 5148 ovens still capable of operating.

Beehive Ovens in the United States as of December 31, 1959

State	Plants in Existence	Ovens			
		In Existence	In Operating Condition	Not in Operating Condition	Abandoned
Pennsylvania	35	6,080	4,125	1,955	1,425
Virginia	5	663	626	37	—
West Virginia	4	512	204	308	—
Kentucky	1	193	193	—	—
Total	45	7,448	5,148	2,300	1,425

For the Connellsville area, once famous for its burgeoning economy, the virtual elimination of the beehive oven as an effective coke producer has been especially disastrous, and the communities of Fayette and Westmoreland counties have for many years comprised one of the most economically distressed areas in the eastern United States. Dominated by the fortunes of a single industry, the Connellsville region had few attractions to bring new enterprises into the district when the beehive ovens were abandoned, and the extent to which the area suffered has not always been realized. Between 1865 and 1918 the Connellsville district produced practically all the coke used in western Pennsylvania and eastern Ohio, and supplied more than 80 pct of that needed by the iron and steel industry in the rest of the country. In

Abandoned beehive ovens to the southwest of Latrobe, Pa.

1916 more than 40,000 ovens carbonized 33,792,256 tons of coal to produce 22,489,056 tons of coke, which was being railed out at the rate of some 25,000 carloads daily. After 1880, rather more than three out of every four workers in the region were employed by the mines and coking establishments, and it was estimated that during the first 10 years of this century the coal and coke interests owned more than 80 pct of the capital investments in the area. The population of Fayette County rose from 58,000 in 1880 to 188,104 in 1920, while in Westmoreland County an increase from 78,036 to 273,568 was recorded for the same period. Between 1920 and 1940, however, Fayette County increased in population by only 12,895. In the latter year, of the 57,390 men in the county available for work, only 38,658 were actually employed. Of the remainder, 5834 were on public emergency work, and 12,898 were seeking jobs. Between 1938 and 1951 employment in the mines of Fayette County went down from 19,639 to 11,965, in Westmoreland County from 10,253 to 7046. A further mirror of the times is provided by the population figures given in the census reports of 1940 and 1950. In 1940 there were 200,699 people in Fayette County; by 1950 the number had dropped by 5.8 pct to 189,-899. In the case of Westmoreland County a 3.2 pct reduction took place, the 1940 and 1950 populations being 303,411 and 293,859, respectively. In view of the unlikelihood that there can be more than temporary and minor resurgences of the demand for beehive coke, it seems likely that this is a trend which will continue.

Epilogue

In the 120 years which have passed since John Taylor built his two crude ovens by the banks of the Youghiogheny, the coke industry has made an immeasurable contribution to the comfort, convenience, and security of life in the United States. Like the smelting of iron and the making of steel, the carbonization of coal has been essential to our well-being in peace and vital to our very existence in war.

What of the future? There can be no doubt that in the years to come this great industry will continue to serve America well. Little publicized, unconcerned with the aura of glamor which has attended the

growth of more recent industrial enterprises, the coking industry has yet accomplished much. The endeavors of an industry which in 1959 gave rise to products valued at $1.3 billion should not be underrated. Neither should its capacity for further achievement, and its promise for a bright future.

It has been possible in this article to do no more than remark the most notable features of the coke industry's development, to follow only the broad tide of its progress leaving many of the lesser rivulets and tributaries unexplored. Some day, perhaps, the fully authoritative and detailed history of coke making in the US will be written. It will make a fascinating story.

Slot-Type Coke Ovens in the US as of December 31, 1959

State	Plants in Existence	Ovens in Existence	Annual Coke Capacity, Net Tons
Alabama	7	1,488	7,249,900
California	1	315	1,450,000
Colorado	1	237	985,500
Connecticut	1	70	410,000
Illinois	6	507	2,714,000
Indiana	5	2,191	10,765,200
Kentucky	1	196	1,185,200
Maryland	1	758	4,174,000
Massachusetts	1	108	665,000
Michigan	4	769	4,416,500
Minnesota	3	241	1,083,600
Missouri	1	96	327,600
New Jersey	1	230	1,100,000
New York	3	830	4,529,100
Ohio	14	2,390	12,648,900
Pennsylvania	14	4,133	20,450,300
Tennessee	1	44	264,000
Texas	2	140	832,000
Utah	2	308	1,345,700
West Virginia	4	742	4,281,100
Wisconsin	1	200	570,100
Total	74	15,993	81,447,700
At merchant plants	20	2,249	10,393,800
At furnace plants	54	13,744	71,053,900

Types of By-Product Oven in the US as of December 31, 1959

State	Koppers	Koppers-Becker	Semet-Solvay	Wilputte	Others	Total
Alabama	338	842	180	65	63[a]	1,488
California	—	315	—	—	—	315
Colorado	100	137	—	—	—	237
Connecticut	—	70	—	—	—	70
Illinois	—	177	—	330	—	507
Indiana	340	1,079	120	652	—	2,191
Kentucky	—	—	120	76	—	196
Maryland	—	758	—	—	—	758
Massachusetts	—	108	—	—	—	108
Michigan	—	259	362	148	—	769
Minnesota	65	156	—	20	—	241
Missouri	56	—	—	—	40[b]	96
New Jersey	165	65	—	—	—	230
New York	186	236	180	228	0	830
Ohio	694	762	176	758	—	2,390
Pennsylvania	1,191	1,965	88	889	—	4,133
Tennessee	—	—	24	20	—	44
Texas	—	140	—	—	—	140
Utah	—	308	—	—	—	308
West Virginia	154	514	—	74	—	742
Wisconsin	100	—	100	—	—	200
Total	3,389	7,891	1,350	3,260	103	15,993

[a] Otto ovens.
[b] Simon-Carves ovens.

Furnace and Merchant By-Product Coking Plants for Selected Years from 1913 to 1959

Year	Number of Active Plants		Total Coke Production, Pct	
	Furnace	Merchant	Furnace	Merchant
1913	20	16	73.0	27.0
1918	36	24	73.9	26.1
1930	46	43	73.5	26.5
1931	46	42	64.3	35.7
1932	44	44	53.5	46.5
1933	42	43	60.5	39.5
1934	41	42	62.5	37.5
1935	40	41	67.3	32.7
1940	45	40	76.8	23.2
1945	53	34	78.4	21.6
1950	55	30	81.5	18.5
1951	56	28	81.7	18.3
1952	57	27	81.6	18.4
1953	58	25	85.1	14.9
1954	58	24	87.5	12.5
1955	58	23	87.6	12.4
1956	57	23	86.7	13.3
1957	57	22	88.2	11.8
1958	55	22	87.7	12.3
1959	54	20	87.5	12.5

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EXTRACTIVE METALLURGY OF VANADIUM

The exhaustion or depletion of present sources of vanadium, together with the trend of new uses, is calling attention to problems of vanadium extraction from new sources. This paper surveys the extractive metallurgy of vanadium.

by Blair Burwell

It has recently been stated that very little can be gained from the literaure about the chemistry and metallurgy of vanadium. It has also been said that vanadium processing procedure has developed more as an art than as a science.

Both of these statements are true and give a good introduction of its metallurgy. This is not because of a lack of literature about the chemistry of the element or that vanadium plants are built and operated in a casual manner, but because the chemical behavior of vanadium follows the unpredictable pattern of the transition elements in many of its compounds, and there is uncertainty as to its precise reaction in the acid and alkali concentrations commonly used in the plants.

The art of its extraction has therefore developed as a balance between a variable chemical behavior and the operation control of the range of variables encountered on each individual circuit. In this problem, the art of observation becomes the science of application.

The chemistry of vanadium is also reflected in the broad range of its occurrences. It enters into life processes of certain micro-organisms in sea water from which it is concentrated and preserved in phosphate beds, limestones, and petroleum resides. Through cycles of oxidation, erosion, and solution of organic tie-ups, many of our important deposits have been derived. These include the Peruvian patronites, lead vanadates, and probably the uranium-vanadium deposits of the Colorado Plateau.

It is an important primary constituent of titaniferous magnetites and iron ores where it is recovered as a part of pig iron production, and in small quantities, occurs widespread in clays and chromites.

Because of its active chemistry in forming subordinate compounds with lead, iron, uranium, or phosphorus, its metallurgy is usually involved in the recovery of other metals.

The end uses of vanadium also reflect its active chemistry and four valencies in use as a catalyst in the chemical field. It performs a vital janitor service in the alloy field reacting with nitrogen or other impurities to form compounds which do not weaken crystal structures. In small quantities, it makes possible the use of tungsten in steel by relieving the brittleness in high speed steel. It is one of the alloying agents, and possibly an essential one, that has enabled the titanium metal industry to survive its problem of metal cracking failure and stage its recent comeback. As a pure ductile metal, it is finding light weight, high strength, and corrosion resistant uses.

Production and consumption of vanadium is increasing rapidly, principally as a result of new uses developing in the general family of transition elements to which it belongs. This includes molybdenum, titanium, uranium, columbium, chromium, tungsten, and tantalum.

From the viewpoint of extractive metallurgy, the exhaustion or depletion of present sources of vanadium, economic or otherwise, together with the trend of new uses, is calling attention to problems of vanadium extraction from new sources.

Salt roasting

As previously stated, vanadium usually occurs in combination with uranium, phosphorus, iron, carbon, or lead and zinc. The usual procedure in separating it from its ores consists of converting it to a soluble sodium salt by roasting with a source of soda under oxidizing conditions. Usually the lowest cost supply of soda is sodium chloride, although sodium carbonate and sodium sulfate can also be used. Vanadium can also be extracted from its compounds by leaching with hot sodium carbonate under oxidizing conditions. The type of sodium compound formed is a function of the method used in forming it, sometimes called the history of the compound, and has an effect on the procedure used in the subsequent precipitation and recovery from its solutions. The usual sodium compound formed in roasting is a polyvanadate which may be expressed as

$$(x)Na_2O \cdot (y)V_2O_5 \cdot (z)O$$

where x, y, and z vary due to temperature, soda concentrations and pH of solutions. The water soluble salts usually noted in the literature are: Sodium orthovanadate—Na_3VO_4, Sodium pyrovanadate—$Na_4V_2O_7$, Sodium metavanadate—$NaVO_3$, and Sodium hexavanadate—$Na_4V_6O_{17}$. Usually in extractive plants, the compounds are complexes of the foregoing.

Most of the problems in pyro-metallurgy arise from side reactions of soda compounds on associated minerals in the ore. These associated minerals are usually silica and silicates, phosphates, clay-like complexes of calcium aluminum silicates, gypsum, calcium carbonates, iron oxides, and organic matter.

Sodium chloride is selective in its attack on vanadium providing the vanadium is oxidized and the

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carbonaceous material decomposed. An exception is calcium carbonate.

Sodium carbonate is non-selective and forms sodium compounds of silica, phosphorus and alumina which interfere with the recovery of vanadium from its solutions.

Sodium sulfate requires a higher temperature for the formation of sodium complexes and is selective in its attack on vanadium. The higher temperature requirements (usually in excess of 850°C), as well as its relatively higher cost, limit its use to certain iron ores, such as the Finnish Otanmaki titaniferous magnetites.

In the oxidation roasting of vanadium ores, there is usually a formation of water insoluble compounds of vanadium with calcium, silica, iron, and alumina by reaction of sodium vanadate and the bases in the oxidation phase of multiple-hearth roasting procedure. This is a well-studied problem in the recovery of vanadium from Colorado Plateau ores, where substantial data has developed on the mechanism of these reactions.

The usual roasting of Plateau ores with salt involves: 1) a reactive period of approximately 45 min and temperature of 750°C. in which, principally, the vanadium in the ore is converted to sodium vanadate, calcium carbonate to calcium chloride, fine silica in part to sodium silicate; and 2) an oxidation period of approximately 1¼ hr and temperature of 840°C in which chlorine and hydrochloric acid is driven off, $CaCl_2$ converted to CaO, and sodium silicate reduced to silicate or calcium silicate.

The important side reactions include the reaction of calcium carbonate with vanadium to form calcium vanadate ($CaO \cdot 3V_2O_5$), the reaction of fine silica with lime to form $CaO \cdot SiO_2$, and with lime and iron in the solid phase to form $3CaO \cdot Fe_2O_3 \cdot 3SiO_2$.

Complex calcium-iron-magnesium-aluminum silicates are also formed, depending upon the molecular ratio of the bases and the time and temperature of furnace contact. The formation of complex calcium-iron-magnesium silicates reduces the formation of calcium vanadates; the equilibrium of this relationship is a function of the time and temperature of the roasting reactions, the fineness of the silica, and the quantity of iron and magnesium present in the roast.

Another important side reaction is the progressive formation, with increasing temperatures, of acid insoluble vanadium (and possibly to a lesser extent of uranium) which is usually termed *silica tie-up*. This tie-up is increased with an increase in the ratio of iron oxides and alumina, and is probably an iron-aluminum-vanadium and silica complex. As the vanadium combined in this reaction is not recoverable in subsequent acid leaching, the establishment of conditions to reduce the formation of this tie-up is important. The loss of vanadium in acid-insoluble, roaster-reaction products varies from 0.10 to 0.24 pct V_2O_5 in the tailings, or from 5 to 12 pct of the total vanadium thus treated.

The effects of the important bases in the roasting of vanadium ores are summarized in Table I.

In general, an increase in the proportions of iron and alumina in the roast increases the losses of vanadium in silica tie-up. Prolonged roasting with iron salts reduces the extraction of vanadium, after reaching a maximum solubility in 1 hr of contact.

Special roasting with additives for ores high in lime involves not only adding reactive materials (usually pyrite) but also adjusting the temperature and time of roasting. Excessive $CaCO_3$ content is usually balanced by the additions of pyrite to the roast, which assist in the reaction by furnishing iron salts for combination with lime and silica to form silicates, and by supplying SO_2 which converts some of the $CaCO_3$ to the less reactive $CaSO_4$. But the effect of the excess iron so introduced is to form insoluble iron vanadium complexes which increase in quantity with time of contact in the furnace, requiring a reduction in the oxidation period of roasting. Incompletely oxidized $CaCl_2$, resulting from shorter roasting cycle, causes re-precipitation of vanadium in the dissolving step following roasting, which is only partially corrected by sodium carbonate leach solutions for the precipitation of calcium chloride as carbonate. It is difficult to precipitate lime from an alkaline solution containing vanadium without some co-precipitation of vanadium.

Another effect of the necessarily shorter roasting time for *special* roasting with pyrite additions is the resultant large quantity of sodium silicate remaining undecomposed from the reactive period. Subsequent acid leaching of the calcine, following water extraction of water-soluble vanadium, is hindered or prevented by silica taken into solution by acid from undecomposed sodium silicate, which often *jells* or seals the sands in subsequent filtration steps.

Additives other than pyrite have been used in the roasting of high lime ores, principally sulfuric acid which converted the carbonate to sulfate without the roast complications inherent in excessive iron additions. Magnesium addition and alunite also have a beneficial effect, but their use imposes additional costs. Roasting has also been successfully done by sodium sulfate at higher reactive temperatures.

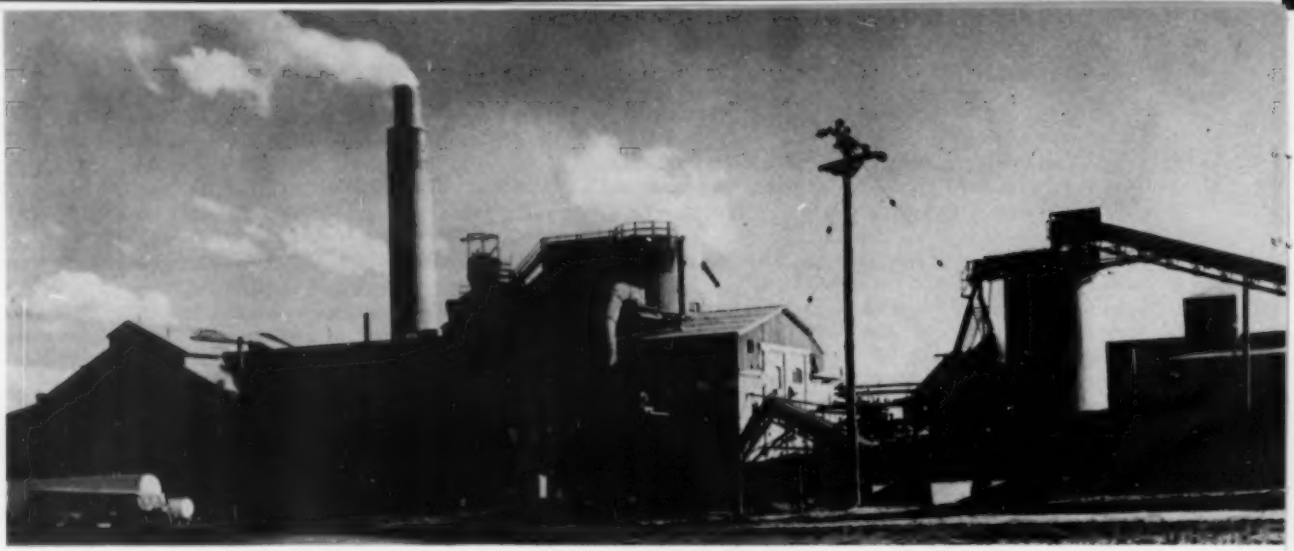
Another reaction is the formation of volatile base metal chlorides, possibly calcium chloro-vanadate, by the action of chlorine in the roast, which are lost in the stack gases. This loss will vary with the temperature conditions of the roast and ratio of bases, but it is probably not less than 1.5 pct of the metal content of the ore. This is apparent when water scrubbing is used on roaster gases, in which case a significant recovery is effected of volatile vanadium and uranium.

Solution reactions of water on the roasted ore, resulting from variations in roaster practice, are important. In combination with losses resulting from the physical conditions of the roast and the cooling of the calcines, they explain much of the debated differences between *extraction* and actual plant recovery.

Plant leaching solutions, which vary in temperature from incoming cool water washes, contain lime in solution in equilibrium with the temperature. This lime is precipitated with temperature increases from

Table I. Effect of Important Bases in Roasting of 2 pct V_2O_5 + 84 pct SiO_2 + 8 pct NaCl at 840°C.

Base	Extraction, pct 1 Hr	Extraction, pct 2 Hr	Silica Tie-up, pct
None	100.0	100.0	0.0
1.2 pct Fe_2O_3	99.4	92.0	7.0
3.5 pct Al_2O_3	87.8	94.7	5.0
1.5 pct CaO	36.0	65.7	0.0
1.0 pct MgO	99.3	97.0	4.0
1.2 pct Fe_2O_3 + 1.5 pct CaO	39.0	71.5	3.0
1.2 pct Fe_2O_3 + 1.5 pct CaO + 1.0 pct MgO	68.0	86.3	4.0
1.2 pct Fe_2O_3 + 3.5 pct Al_2O_3 + 1.0 pct MgO	88.6	87.3	12.0



contact with hot calcines or soda ash used in the circuit. Vanadium usually co-precipitates with lime under these conditions and increases tailing losses. A general metallurgical tabulation of results from salt roasting follows:

	Low Lime	High Lime
	Normal Salt Roasting	Special Salt Roasting
Recovery V_2O_5, Roasting Only, pct	74	65
Recovery, Roasting and Soda Ash Leaching (Quench), pct		76
Recovery, Roasting and Acid Leaching, pct	83-90	85-90
Silica Tie-up Losses, pct	7-12	5
Approximate Maximum Temperature, °C	840	900

Acid leaching

Up to 1945 the recovery of vanadium from Colorado ores dictated plant installations and recovery systems. In the main, they were built around multiple-hearth roasting furnaces and produced a high soda *red cake* and fused oxide as a standard product. Beginning in 1943, the recovery of uranium became more important than vanadium, and original vanadium roasting and leaching plants were modified by the addition of acid leaching to tails from the vanadium recovery circuits. Soda roasting for vanadium does not attack the uranium content of the ore. It does provide a calcine for subsequent acid leaching which is readily filterable. As the relative importance of vanadium to uranium recovery continued to diminish, and at the same time higher grade vanadium ores were depleted, the direct leaching of ores by sulfuric acid to extract uranium replaced, in part, primary roasting for vanadium recovery. To avoid a difficult filtration problem from the colloidal slimes of direct acid leaching circuits, the Atomic Energy Commission recently sponsored the development and application of the *resin in pulp* method of ion exchange recovery of uranium. This, together with the following solvent extraction on strong acid leach solutions, has become the standard metallurgy of uranium recovery in new plants in the region.

Acid leaching of uranium ores, if carried out at acid concentrations required to obtain high recoveries of uranium in extract liquors, also results in high iron, silica, and phosphorus contents in the solution. While these do not seriously interfere with the recovery of uranium, they impose difficulties on related vanadium recovery. Up to 1958, the vanadium-bearing acid tail liquors for uranium recovery were mostly wasted or impounded in western plants. In 1958, the Bureau of Mines developed a solvent extraction method of recovery of vanadium from these solutions, and other operators, notably Union Carbide Corp., carried on similar developments.

The application of solvent extraction of vanadium from uranium acid leaching plants varies from plant to plant in detail.

At the Shiprock plant of the Kerr-McGee Uranium Co., the sulfuric acid tail liquors from uranium extraction containing vanadium in the reduced valency (VO_4) are loaded on an organic solvent at a pH of 1 to 2. The organic is then stripped with 5 pct sulfuric acid. The strong acid strip liquor is then stripped of its vanadium by heat and oxidation, yielding vanadic acid, HVO_3, or *acid red cake*. As the acid strip liquor contains iron and phosphorus, the recovery of vanadium oxide is carried on at a high acidity which results in the phosphorus and the largest part of the iron remaining in the acid raffinite. This is fed into the uranium leach circuit carrying the unprecipitated V_2O_5. A portion of the V_2O_5 then recycles through the uranium leach circuit.

A similar circuit is in operation at Edgemont, S.D., at the plant of Susquehanna-Western, Inc.

The recovery of vanadium from its solutions can be divided into two general methods depending on the purity of the vanadium compound desired. The first is precipitation as *red cake*. The term red cake is applied to a variety of deep red hydrated complexes of vanadium with a wide range of soda content, from none to approximately 15 pct, and with approximately 75 pct of moisture. When dried and fused, it becomes the standard *fused oxide* of commerce and the conventional source of vanadium for ferrovanadium.

Red cake precipitation is usually applied to the recovery of vanadium from soda solutions resulting from salt roasting. The precipitation of red cake from these solutions is easier and more complete than from soda solutions of other origin. Part of this is due to the lower solubility of the soda complexes in the salt-bearing solutions from the roast, and in part, to reactions in the salt roast which assist in the

Left, Climax Uranium Co.'s plant at Grand Junction, Colo. Courtesy of Climax Uranium Co.

Right, Union Carbide Nuclear Co.'s plant at Uravan, Colo. Courtesy Union Carbide Corp.

formation of higher sodium-vanadium aggregates requiring less time for red cake formation.

Usual solutions from roasting are slightly alkaline. For standard red cake—more than 86 pct V_2O_5 and 9 to 10 pct Na_2O—the solutions are heated to boiling and concentrated H_2SO_4 added to a pH between 2 and 3. As acid is added, a red color develops of a colloidal vanadium complex followed by hydration and agglomeration as heating and acidification proceeds. A *rolling* agitation is used with a definite peripheral speed to form round nodules which can be filtered. There is a definite *seeding out* action in precipitation, which is assisted by the quick formation of an initial precipitate, and a fast addition of acid. Low grade solutions below 5 gpl are difficult to precipitate and require long periods of boiling due in part to the lack of nuclei.

Organic matter and reduced vanadium inhibit or prevent the precipitation of red cake. Phosphorus also affects the precipitation. If present in excess of 1 gpl, it decreases recovery from solutions from 99.5 to 85.0 pct.

Low soda red cake is formed by a variation in the manner of acid additions, and by the introduction of ammonia salts into the precipitating liquor. When sulfuric acid is added to a cool alkaline soda roast solution, a vanadic acid is formed low in soda. This compound, when heated, hydrolizes and forms a red cake low in soda, usually less than 3 pct. The addition of ammonia salts to the solution also assists in reducing soda by replacement with ammonia.

A second method of precipitation of vanadium from alkaline soda leach liquors is by the use of ammonium chloride. When ammonia, or ammonium chloride, is added to a sodium vanadate liquor, soluble ammonium metavanadate (NH_4VO_3) is formed. The solubility of this compound decreases with an increase of concentration of excess ammonium chloride in the solution. The occurrence of sodium chloride in the solution also affects the solubility. With a concentration between 85 and 100 g of ammonium chloride in the solution, the precipitation is practically complete.

Ammonium metavanadate, when ignited, yields pure V_2O_5 from the elimination of ammonia, and this method of precipitation is used to obtain pure oxide required in the manufacture of catalysts and pure metal and alloys. As the tail liquors from such precipitation methods contain a large excess of ammonium chloride, recovery methods for re-use of the reagent are necessary. These are usually accomplished by evaporation and crystallization on cooling.

Recovery from phosphate ores

Western phosphate ores constitute an important and little used source of vanadium. The vanadium content varies from 0.10 to 1.0 pct V_2O_5.

Prior to 1950, the Anaconda Copper Co. recovered approximately 250,000 lb of V_2O_5 annually as standard red cake (86 pct V_2O_5 and 9 pct Na_2O) as a part of their Anaconda tri-super-phosphate production.

In this process, calcined ore is leached with sulfuric acid, and the resulting filtered phosphoric acid solution concentrated by evaporation. The V_2O_5 present in this acid solution is precipitated as an impure hydrated sodium vanadate by oxidation with sodium chlorate. By re-dissolving with soda ash and re-precipitation at a low pH, a sodium vanadate precipitate low in phosphorus results, which is dried and used to form a soluble product.

With the increasing use of western phosphate ores for elemental phosphorus recovery, substantial quantities of vanadium are now concentrated in the ferrophosphorus tappings from electric furnace reduction and smelting of ores. The iron content of the ore acts as the gathering metal for the small vanadium and chromium content producing a ferrophosphorus with 4 to 6 pct V. The recovery of vanadium here is complicated by its relationship to phosphorus, chromium, and iron. When soda roasting is employed to solubilize the vanadium, sodium phosphate, and sodium chromate are also formed. The separation of these soda salts has, so far, not reached the stage of plant application although selective crystallization (Bureau of Mines process) and solvent extraction has received extensive study.

A new plant, involving a different principal, is scheduled to go an stream in Salt Lake this year. So far, this metallurgical step is not available for publication.

Recovery from iron ores

Vanadium occurs in small amounts in many iron ores, especially in certain magnetite ores of Sweden and Finland and in German ores. The average vanadium content of this ore is: Swedish iron ores—0.1 to 0.25 pct V, Minette ore—0.07 pct V, and Watenstedt ore—0.20 pct V. During World War II, German steelmaking recovered up to 300 tons of ferrovanadium monthly by treating these ores for vanadium recovery and survived the allied economic blockade by using vanadium so produced in place of molybdenum and tungsten. While this production was not economic in normal times, it has stimulated attention to the recovery of vanadium from titaniferous magnetite deposits in many parts of the world. Due to the magnitude of these reserves, they constitute a major potential supply.

In the past fives years, two new sources of vanadium have developed in plants in Finland and the Union of South Africa, treating titaniferous magnetites and recovering high purity V_2O_5 as fused oxide. So far the utilization of these ores has been confined to a V_2O_5 content in excess of 0.60 pct.

The MECSA plant at Witbank, South Africa, treats a titaniferous iron ore from selected high grade ores containing a minimum of 1.5 pct V_2O_5. These ores contain: 50.0 to 55.0 pct Fe, 10.0 to 12.0 pct TiO_2, 1.2 to 1.75 pct V_2O_5, 1.2 to 5.0 pct SiO_2, and 1.0 to 2.0 pct CaO.

Vanadium probably exists in solid solution with the iron as $Fe_2O_3 \cdot VO_2$. Upon oxidation roasting with salt, a thermal decomposition of the iron complex occurs with the formation of Fe_2O_3 and the release of V_2O_5 to combine with sodium, forming sodium hexavanadate.

The multiple-hearth furnace practice at this plant follows the practice on the Colorado Plateau. The ore is ground to −60 mesh, mixed with 8 to 10 pct salt, and roasted at a temperature of 800°C for approximately 1 hr. The calcines are cooled and leached in perculation tanks.

A particular feature of this plant is the recovery of V_2O_5 by precipitation as a high grade oxide (100 pct V_2O_5) by the use of ammonium chloride in leach solutions and the recovery and re-use of ammonium chloride and salt. A combination of cheap fuel and labor plus costly salt and chemicals dictates this metallurgy.

The first vanadium plant in northern Europe, the Otanmaki plant in Finland operated by the Finnish government, started operations in 1955. It has since been increased in capacity to approximately 2 million lb of V_2O_5. The ore is the government-owned, magnetite-illmenite deposit which contains, in the crude ore, approximately 0.40 pct V_2O_5. The metallurgy employed here is the result of the need of recovery of a magnetite concentrate reasonably low in titanium, an illmenite concentrate, and vanadium oxide. As is typical of this type of ore, the vanadium follows and is associated with the iron. The following is gained from an article by M. H. Tikkanen, *The Extraction of Vanadium from Vanadium-Containing Magnetite Iron Concentrate*, and from visits of engineers to the operation.

The crude ore, as mined, contains approximately 0.40 pct V_2O_5. It is concentrated by a combination of wet and dry magnetic concentration into an illmenite fraction and a magnetite fraction containing approximately 68 pct Fe, 2.2 pct TiO_2, and 1.0 pct V_2O_5. This concentrate is finely ground and pelletized with sodium sulfate, forming pellets which are fed into a high temperature (1200°C) shaft furnace. This step is apparently designed to produce an iron pellet for sale to the steel industry and to solubilize the V_2O_5 at the same time. Due to the size of the pellets, contact time is long (12 hr). The soluble sodium vanadate is leached from the pellets and precipitated by sulfuric acid and heat at a low pH (2 to 3) with oxidizers to form acid red cake and fused oxide. The pellets are dried and shipped as feed to the blast furnace.

This plant is a well worked out composite of metallurgical economies and illustrates, in general, the straddle which usually exists in extractive vanadium metallurgy. The well-known problems of pellet formation, burning and heat percolation are combined with high temperature soda roasting, oxidation and leaching.

Recovery from organic-sulfur combinations

The typical extractive metallurgy of this source is probably Minasragra deposits of Peru, which up to 1930, supplied the largest part of V_2O_5 for world supply. This deposit is credited with being the source of early vanadium supply which introduced the metal to world use before 1920.

The first, and major part, of the recovery of vanadium from this source was not by extractive hydrometallurgy, but by direct electric furnace reduction of high grade ores (20 pct V_2O_5 or better) producing the *open hearth* grade, direct reduction ferrovanadium, which contains from 30 to 40 pct V. As high grade, direct reduction ores were depleted and markets developed for ferrovanadium of higher vanadium content, extractive metallurgy was applied by roasting, leaching, and red cake precipitation on low grade ores. The property is now inactive.

Recovery from lead and zinc vanadates

This important source of vanadium has long been a by-product of lead and zinc production from African mines (Broken Hill in Northern Rhodesia and Abenab and Tsumeb in Southwest Africa). The vanadium occurs principally in the oxidized surface zones of lead-zinc deposits as descloizite (lead and zinc vanadate). Ores of Southwest Africa have long been concentrated at the mines and shipped to European reduction works where lead is recovered as metal by smelting and vanadium extracted as sodium vanadate by sodium carbonate roasting and recovered as red cake.

Outlook

Due to the great variety of mineral associations, it is difficult to predict where the source of vanadium for the future will be produced. The indications are that the Colorado Plateau source is substantially depleted by its contribution of 60 pct of world supply and cannot be operated as a source of vanadium without an active uranium industry. What will happen here after 1966 when uranium purchases by the Government expire will determine the availability of this major supply. It looks as if the active time for survival research is in the making.

LATIN-AMERICAN IRON AND STEEL CONGRESS, MARKS BEGINNING OF NEW ERA

Robert W. Shearman, Secretary of The Metallurgical Society, attended the first Latin-American Iron and Steel Congress as an "Observadore Invitado", representing AIME. The trip was sponsored by the Electric Furnace Committee. This article describes that meeting. At a later date, JOURNAL OF METALS *plans to feature articles on the economy and steel production of the Latin-American countries.*

by Robert W. Shearman, Secretary
The Metallurgical Society of AIME

Steelmaking in Latin America has come of age. The First Latin-American Iron and Steel Congress was held in Sao Paulo, Brazil, May 2-5, under the auspices of the 18-month old Latin-American Iron and Steel Institute (Instituto Latinoamericano del Fierro y el Acero), commonly known as "ILAFA." The Congress provided opportunities for technical sessions, business meetings, and plant trips. Perhaps most important of all—the Congress became *the* forum for steelmakers from all the steel producing countries of Latin America to meet each other, to become friends, and to exchange information for the common good. ILAFA was founded in Santiago, Chile, in the fall of 1959 by representatives of the nine countries that produce iron and steel in Latin America.

On the agenda for discussion by the First Congress were these important items:

1) Establishment of a free trade zone for iron and steel within a Latin-American Common Market;
2) Examination of laws and regulations of Latin-American countries that affect production of iron and steel;
3) Development of a common vocabulary of technical terms;
4) Standardization of iron and steel products; and
5) Devising a means of statistical reporting.

Common-Market principle

General recognition was given to the need for developing a free zone for iron and steel in Latin America. The treaty establishing a Common Market had just been signed in Montevideo as the Congress was convened. Great hope was expressed that ILAFA could spearhead the development of the Common Market, since it represents the only trade association organized to date in Latin America.

However, it was also recognized that the problems in Europe that led to the creation of the European Coal and Steel Community and later to the European Common Market, were far different from the problems in Latin America. The European participants were countries exporting steel; Latin America is an importer of steel, and will remain so for many years. Present production is below 5 million tons per year and consumption about 8 million tons. For the 15 million-ton expansion in steel capacity planned, capital requirements will approximate $4500 million. Since capital is the crying need in Latin America, the objective of a Common Market in steel could well become the reduction of capital requirements through location of expensive plants at strategic locations, and available to more than the *host* country alone.

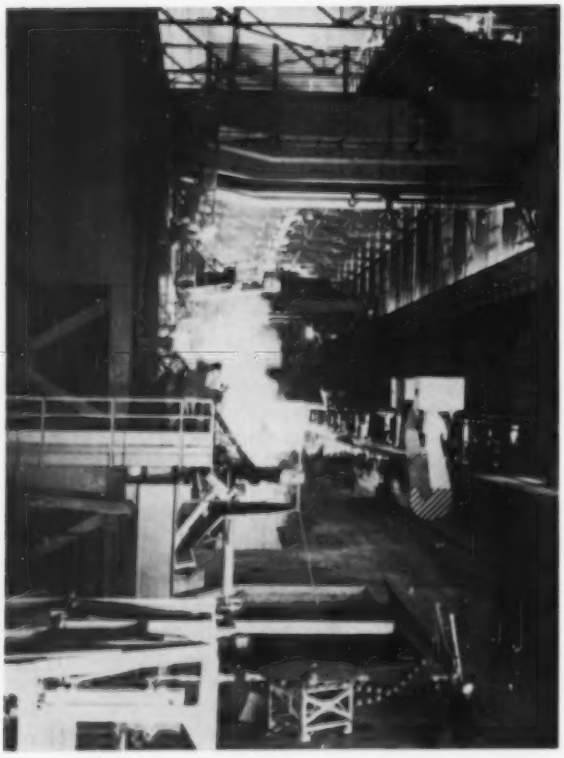
Headquarters in Chile

Although some progress was made in examining the other items on the agenda, there was one important administrative matter that overshadowed and took priority—where to locate the permanent headquarters of ILAFA? This problem was one for the smoke-filled rooms, but its importance was recognized whenever two Congress registrants stopped to chat. Finally, at the concluding General Assembly on May 5, national pride was put aside and the temporary headquarters in Santiago, Chile, were declared the permanent headquarters.

The technical side

Latin America is blessed with high-grade iron ore in abundance. Unfortunately, high-grade coking coals are lacking. Hence, new metallurgical processes using non-coking coals, natural gas, and oxygen are being studied. Cheap hydroelectric power for direct reduction processes offers possibilities. Charcoal

General Edmundo de Macedo Soares e Silva, President of ILAFA, addresses the opening session of the First Latin American Iron and Steel Congress.



The first open-hearth heat is poured at the General Savio plant of SOMISA, San Nicolas, Argentina.

blast furnaces, using the wood of the eucalyptus tree, are prevalent in Brazil.

With these factors as a background, particularly the lack of coking coals, Marc Allard, director of IRSID in France, presented a technical paper describing development work on the use of low-grade Brazilian coal for metallurgical coke at the Liege low-shaft blast furnace. Joining in the discussion were Jacques Astier, also of IRSID, and Pierre Coheur of CNRM, Belgium. Session chairmen were Roberto N. Jafet, technical director of Mineraco Geral do Brasil, and Renato Frota Rodriguez de Azevedo, industrial director of Companhia Siderurgica Nacional at Volta Redonda, Brazil.

Jack R. Miller, president of the consulting firm, J. R. Miller Co., New York, talked on the need in Latin America to consider the economics of processes in addition to technical aspects. Thus, to increase steel production in Latin America, and to combat the problem of lack of coking coal, he recommended consideration of a specially-designed blast furnace that would operate on 100 pct sinter. Comparable capital costs were 0.87 for the sinter furnace vs 1.00 for the classical blast furnace. For steelmaking, oxygen must be considered, with emphasis placed on lower capital investment, particularly for open-hearth roof lances. For electric furnaces (which, in Mr. Miller's opinion can do anything the open-hearth furnace can do), the most economical production rate is about 500,000 ingot tons per year.

During the Congress, the stimulating news was received that the first heat of steel had been poured

Left, Luncheon at Binder's Restaurant, in the outskirts of Sao Paulo, between plant trips.

Right, Marc Allard of IRSID, France speaking at a technical session. At left are the session chairmen, Roberto N. Jafet of Mineraco Geral do Brasil, and Renato Frota Rodriguez de Azevedo of Companhia Siderurgica Nacional. At right is Hector Canguilhem of Compania de Acero del Pacifico, Chief of the Technical Dept. of ILAFA.

Below, Fernando Aguirre Tupper, Secretary General of ILAFA, speaking to the General Assembly. Left to right, Cesar A. del Rio of Corp. Peruana del Santa, Peru (2nd Vice President of ILAFA); Carlos Prieto of Compania Fundidura del Fierro y Acero de Monterey, Mexico (1st Vice President of ILAFA); General Soares, President of ILAFA; Sr. Aguirre; and Luiz Coelho Correa da Silva, Regional Secretary for ILAFA in Brazil.

on May 5 at the new integrated steel plant of SOMISA in San Nicolas, Argentina. Planned yearly production from four open hearths is 632,000 tons.

At an evening session on May 3, a talk and motion picture were presented on the Kaldo basic oxygen process by Nils Paues, a representative in Brazil of several Swedish firms.

The following evening, for those not too weary to attend, a talk was scheduled on *Dry Quenching of Coke* by Bernard C. Blanche, Société Carbonisation et Céramique, France. Described was a method of using a combination of nitrogen, carbon dioxide, and steam for quenching. The process is used at the plant of SIDELOR at Homecourt, France. This talk was followed by a motion picture with excellent photography showing the construction in Japan of a new integrated steel plant near Yawata in 1959. Using the LD basic oxygen process, production is now 5.5 million tons per year.

Plant trips

Time out for inspection trips on the third day (May 4)! Busy, industrial Sao Paulo presents no problem in scheduling plant trips. Rather, it is a matter of selection. Bus departure in the morning was for Aco Villares at Sao Caetano do Sul, a suburb of Sao Paulo. Although current production is 36,000 tons per year, planned expansion will soon give production of 160,000 tons per year of alloy steels, special castings, and forgings. This modern plant employs 2100 persons. Visitors were segregated by language requirements, and small groups were led by guides speaking Spanish, Portuguese, or English.

A basic factor underlying the current industrial expansion in Brazil is the introduction of motor cars built in that country. The afternoon plant trip gave the opportunity for a first-hand inspection of this phenomenon. The company visited was Mercedes Benz do Brasil, employing 5400 persons. Trucks and buses are turned out in a very modern plant on a production-line basis. Quality control is stressed. Stock ownership is 50 pct by Daimler-Benz of Germany.

The automobile industry has made a terrific impact on the economy of Brazil. Auto manufacturers under the Kubitchek regime were encouraged to locate in Brazil, but were obliged to obtain an increasing number of their parts from Brazilian firms. The result is the increased need for steel production (and, in turn, the return of more scrap to furnaces). Also, the increased requirement for construction of roads and bridges, is a by-product.

Following the formal closing of the Congress, a plant trip was scheduled to one of the installations of Mineraco Geral do Brasil on Saturday, May 6. During the following week, on Monday trips were scheduled to the Institute of Atomic Energy, Sao Paulo; Companhia Brasileria de Material Ferroviario (COBRASMA), which supplies railroad equipment and is associated with American Steel Foundries; and to Cobrasma Rockwell Eixos (CRESA).

Tuesday May 9, the visit was to General Motors do Brasil. And Wednesday, was devoted to an inspection tour of Companhia Siderurgica Nacional at Volta Redonda, which now produces steel at the rate of 1.2 million tons per year.

In spite of a full schedule, the social man was not neglected. Prior to the formal opening session, on the evening of May 2, a gay reception and buffet dinner were extended to all delegates by the Brazilian Directors of ILAFA. With wives in attendance, food, beverages, and conversation were in abundance in the roof garden of Sao Paulo's *Jardim de Inverno*.

A gala *Comida de Despedida* (Fellowship Dinner) at the Othon Palace Hotel brought the formal Congress to a close on Friday evening, May 5. With excellent Brazilian food, refreshments, and entertainment, and with pleasant companions, "a good time was had by all."

For those taking the Saturday plant trip, the climax was a *churrasco* (barbecue) at the home of Roberto N. Jafet, Brazilian Director of ILAFA.

The men who make ILAFA

Head of ILAFA is its President, General Edmundo de Macedo Soares e Silva, who is credited with conceiving and pushing to completion the successful steel plant at Volta Redonda, Brazil. Retiring recently as President of Companhia Siderurgica Nacional, he is now President of Mercedes Benz do Brasil.

In the important post of permanent Secretary General is Fernando Aguirre Tupper from Santiago, Chile, the man who makes ILAFA function.

The man whose responsibility it was to plan the successful Sao Paulo Conference was the Brazilian Regional Secretary, Luiz Correa da Silva, who is also a Professor of Metallurgy at the University of Sao Paulo.

First Vice President of ILAFA is Carlos Prieto, President of Compania Fundidora del Fierro y Acero de Monterrey, Mexico. Second Vice-President is Cesar A. del Rio, Director of SOGESA at Chimbote, Peru. Representatives of other steel producing countries serve as directors.

All officers and directors were re-elected to serve one more year in office.

The registrants

About 125 registrants attended the Congress—representing all the steel-producing countries of Latin America except Cuba.* Included were men in management, production, and research and development. Foreign countries sending representatives were the United States, France, Belgium, and Japan. Registrants from the United States included Carl Cass, Chief Engineer of the Export-Import Bank, Washington; Rafael Suarez-Guzman, Interamerican Bank for Development, Washington; Jack R. Miller, Consultant, New York; Michael A. Wrotniak, Jr., Great Lakes Carbon Corp., New York; and the writer.

Optimism was the mood of the Congress at adjournment, in spite of serious technical and economic problems yet to be faced. Cooperation between companies and between countries will help solve many of these problems.

The Second Congress of ILAFA will be held in Buenos Aires, Argentina, the first week of April, 1962—just time enough for a registrant to return to the United States for the National Open Hearth Steel Conference in Detroit, April 9-11. All aboard!

* Argentina, Brazil, Colombia, Chile, Mexico, Peru, Uruguay, and Venezuela.

First International

AGGLOMERATION SYMPOSIUM

A summary of the First International Symposium on Agglomeration held in Philadelphia, Pa. April 12-14, 1961.

All phases of agglomeration—sintering pelletizing, nodulizing, briquetting, powder metallurgical techniques, ceramic bonding, and related processes—were considered at the First International Symposium on Agglomeration held in Philadelphia's Sheraton Hotel, April 12-14. And an international symposium it was, with over 595 registrants from more than 16 countries. Technical information from 11 different countries was disseminated from the 35 papers presented during the three-day symposium.

In addition to the technical competence of the meeting, several other features should be emphasized. At the two informal luncheons, each author was requested to sit at a particular table displaying his name, thus affording an opportunity for interested people to dine informally with the author. Not only did this procedure offer an excellent chance to meet the various authors personally. but it also provided time to discuss particular papers. Another excellent feature of the symposium was the availability of a complete set of preprints to each registrant. For those registering in advance, a set of preprints was posted several weeks before the conference, thereby providing an opportunity to study papers prior to the meeting; this assured a stimulating discussion at each conference session.

Welcoming luncheon

AIME President R. R. McNaughton, welcomed the international audience with a talk entitled, *Risk of Failure is the Price of Progress.* President McNaughton stated that, "The object of science is knowledge; the object of art is works. Science inquires *Why.* Engineering, the modern version of ancient practical arts, asks *How* . . . The scientist finds his inspiration in the rewards of discovery resulting from the quest for further knowledge of nature and nature's laws. The engineer's approach is quite the reverse. His is the creative viewpoint. His is the task of devising, planning, and providing the means of effectively meeting the material needs of civilized life . . . He is the *doer,* spurred on by the creative instinct in man but laboring under the limitations imposed by material, human, and technical resources at his command and the economic and social demands of his day. . . Like the man of medicine, he often cannot wait for full or complete understanding before acting. The risk of failure is the price of progress.

"The role of engineering in providing for man's material needs is as old as civilization . . . Yet only recently have historians turned their attention to the vital role which engineering plays in the progress of Western civilization and in creating the world in which we live today . . . Indeed the engineering advances of the last century or two have almost completely changed not only the world in which we live but the fundamental relationships of men. . . In sharp contrast to the largely independent, largely self-sufficient farm-house economy of a century ago, no man in the western world stands alone today. We are dependent on our fellows not only for convenience, but for the very basic needs of life . . ."

"The major problem the Western world faces today is to achieve . . . co-operation within the sphere of liberty, free enterprise, and private initiative, the conditions under which . . . advances originated and have been nourished and developed."

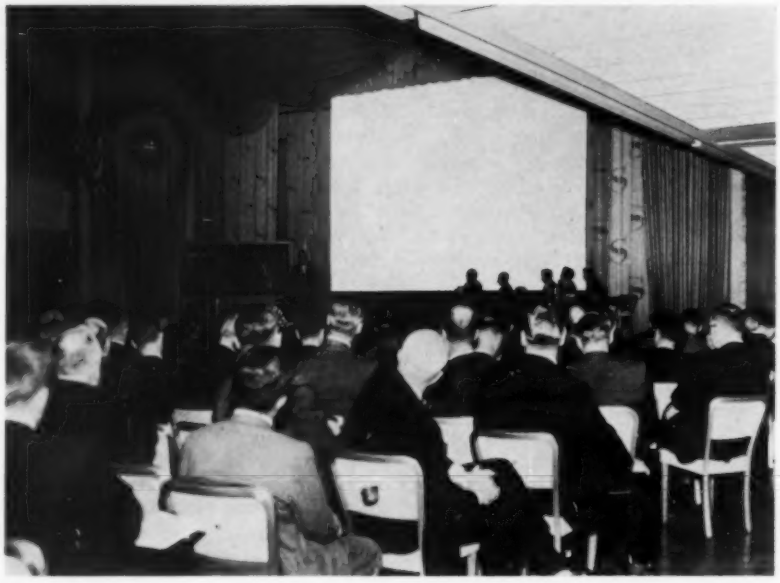
"Our only hope, not only of supporting populations that are ever increasing the world over but of providing higher standards of living for underdeveloped countries, rests in continued scientific and engineering advances."

Technical Sessions

Summaries of the papers thought to be of particular interest to JOURNAL OF METALS readers follow.

An Evaluation of the Properties of Dolomites Affecting Self-Fluxing Sinter Rates, R. A. Limons and H. M. Kraner, Bethlehem Steel Co., described the experimental production of self-fluxing sinter on a laboratory scale. Several dolomites from Ohio, Michigan, and Canada were employed. Chemical

The Welcoming Luncheon, shown at the left, was the only formal luncheon of the symposium. The other luncheons were informal and provided the registrants an opportunity to discuss papers with individual authors.



Right, a technical session. One of the seven sessions at which a total of 35 papers were presented.

analyses of the dolomites were essentially the same varying slightly as follows: 30.9 to 31.6 pct CaO, 21.8 to 22.2 pct MgO, 0.18 to 1.00 pct Si, 0.08 to 0.72 pct Al_2O_3, and 0.06 to 0.15 pct total alkali. Additions of approximately 15 pct dolomite were made to the sinter mixes to insure that the base-to-acid ratio would be slightly greater than 1.0. The raw materials were mixed in a muller type blender and then charged into a 9-in. diam cast-iron sinter pot having 10-in. high walls. The mix was ignited for 30 sec using a fixed air-to-gas ratio; initial vacuum averaged 30 in. of water. Sintering was continued for 4 min after which the sinter was discharged and weighed.

Results indicated that screen size and average crystal diameter of the dolomites are properties having a major effect upon sinter rate and strength.

Sinter mixes which contained 15 pct dolomite screened to −8 +10 mesh produced the highest sinter rate; however, sinter production rates differed as much as 0.75 ton per sq ft per 24 hr among mixes containing the different dolomites even when the dolomite additions were screened to the same size.

Petrographic examinations showed a variation from 0.0025 to 0.0075 in. in the average crystal diameter of the dolomites. Also, it was found that dolomites composed of fine crystals evolve their CO_2 more rapidly at high temperatures than do dolomites composed of coarse crystals. Thus, the available reaction time between the CaO and MgO of the dolomite fines and the iron oxides and gangue of the sinter mix is greater when fine crystal dolomite is used than when coarse crystal dolomite is used. It was shown that dolomites composed of fine crystals produced higher sinter rates than dolomites composed of coarse crystals.

To observe the variations in self-fluxing sinter rates, a laboratory apparatus was developed which permitted the visual study of the downdraft sintering process. The sinter mix container was a pyrex glass tube 16 in. long, 4-in. in diam, and a ⅛-in. wall thickness—rather than the usual pot. Several interesting phenomena were observed during runs in this apparatus. For example, segregation of coarse

and fine particles occurred during charging; the sinter mix compacted as much as 1½ in. when suction was applied before ignition; moisture condensed on the container walls and on individual particles during ignition and throughout sintering; the flame front advanced in an irregular fashion; calcination took place immediately below the flame front; fines were drawn through the grate as sintering neared completion; and additional shrinkage took place until sintering was completed.

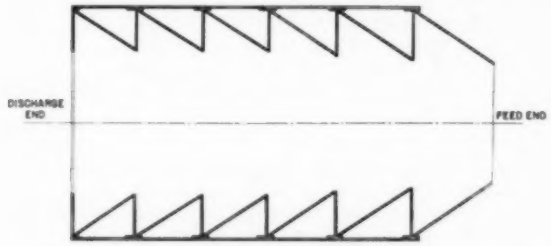
It is believed that, by controlling the screen size and crystal diameter, higher, more uniform production rates and better quality of self-fluxing sinter can be obtained.

Various types of revolving machinery are used to roll and compress fine material into small balls for sintering or into pellets to be hardened. H. T. Stirling, Koppers Co., Inc. described a novel type of balling drum in his paper on *Advances in Balling and Pelletizing.*

The author pointed out that in order to join fines together successfully by balling or pelletizing, the many operating variables must be adjusted to the most desirable value if optimum results are to be obtained.

In order to provide higher retention times and better rolling and compression action than disc-type and standard rolling drums, a multiple-cone drum was designed after many months of trial and error testing.

The drum, shown schematically in the accompanying figure, contains a series of compartments each segregated by an annular baffle. Each cone forms an angle of about 34° with the edge of the drum. This design permits superior rolling action to that occuring in a cylindrical balling drum while to a great extent eliminating the free travel period that occurs in a disc during which the balls or pellets are carried up without any rolling. Furthermore, the baffled compartments tend to hold back larger quantities of material than is possible with a cylindrical drum, thus providing a longer retention time than with a cylindrical drum of equal size.



Because of the way the material is slightly wedged into the bottom of each bay as it travels upward, the multiple-cone drum does not subject each particle to the high centrifugal force necessary to prevent slipping in a standard drum; therefore, the peripheral speed of the multiple-cone drum is about half that of a standard drum. In addition, the multiple-cone drum acts similarly to a disc in that it tends to classify material, holding back the fines while the formed balls or pellets pass into the next bay. Comparison of the sintering rates obtained from several mixes processed through the multiple-cone drum with those obtained from the same mixes after preparation in a pug mill, disc pelletizer, or standard balling drum indicate an increase in rate when the multiple-cone drum is used. A large increase in the sintering rate is usually obtained when the return fines are added to the mix after balling. It was also found that fly ash and grindings from plate-glass factories can be successfully balled for sintering into lightweight aggregate in the multiple-cone drum using about 20 pct water with no other binder.

Hot Briquetting of Partially Reduced Iron Oxide Ores and Dusts, by J. E. Moore and D. H. Marlin, Dravo Corp., was a presentation of the results of studies of a method for agglomerating iron ores, ore concentrates, and iron oxide dusts by partial reduction and briquetting at temperatures up to 1500°F.

Tests during this investigation indicated that most natural hematite or magnetite ores could not be satisfactorily briquetted under the pressures employed at temperatures below 1300°F. However, when the ores were partially reduced, the strength and integrity of the briquets markedly increased. The lower oxides that were formed produced stronger bonds than natural hematite or magnetite when pressed in the range of 700° to 1500°F. Microscopic examination and microhardness tests indicated the hematite particles are the hardest; natural magnetite is significantly softer than hematite; artificial magnetite is slightly softer than natural magnetite and significantly softer than the hematite from which it was reduced; and wustite is the softest of the oxides. At elevated temperatures, the relative hardness of the various oxides are probably such that Fe_2O_3 is the most brittle and wustite the most ductile.

In the briquetting procedure, hot, prereduced ore was force fed into the briquetting machine, and the briquets were discharged at the bottom. The forces between the 2-in. wide rolls were adjusted up to 68 tons, and briquetting rates of 500 to 2000 lb per hr were employed. The following factors were found to affect directly the chemical and physical properties of the briquets: 1) raw material characteristics, 2) preheating and partial reduction prior to briquetting, 3) briquetting conditions, and 4) treatment after briquetting.

The briquetting process is not limited to any specific size range, although stronger briquets were usually produced from materials having larger amounts of fine particles or broader size ranges. The addition of fine particles to a coarse concentrate greatly increased the briquet strength.

Preheating and partial reduction of the raw material, prior to briquetting, eliminated volatile matter and improved the weldability of the iron-oxide particles. It was found that the location of the various oxides during the instant of briquetting influenced the strength of the bond.

Crushing strength tests revealed that briquets that were rapidly cooled in air or by water sprays and those that were cooled by direct heat exchange with damp ore were of approximately equal strength, but those which were water quenched were weakened because of thermal stresses and cracks which developed during quenching.

Studies of this method for hot briquetting partially reduced iron oxides have shown the process to be applicable for agglomerating a large variety of iron-bearing ores, ore concentrates, and dusts. Chemical and physical tests and microscopic studies have shown that the combination of heat, partial reduction, and pressure causes strong bonds to be formed

between the individual particles within the briquets. Laboratory and pilot plant investigations have demonstrated the feasibility of the process for producing a uniform product of predetermined characteristics. The tests have shown that the size, density, strength, porosity, and chemical state of the briquet can be closely controlled and can be varied within wide limits.

Studies of briquet properties, including reducibility, high-temperature strength, and reduction-disintegration, have indicated the briquets to be suitable for use in iron and steelmaking furnaces. Comparative tests have also indicated that, for a number of possible applications, this process has several potential advantages over other methods of agglomeration.

The relationship between measured properties of zinc oxide sinter and certain performance characteristics of electrothermic zinc furnaces was presented by D. E. Warnes, St. Joseph Lead Company, in his paper, *Properties of Sinter Smelted in the Electrothermic Zinc Furnace*. The author discussed the method of measuring sinter hardness, the relationship of sinter hardness with electrothermic furnace zinc elimination, and presented reasons for the observed relationship between hardness and elimination.

A tumbler test method, described in the 1951 proceedings of the Blast Furnace, Coke Oven, and Raw Materials Committee, was employed to measure hardness. Test work showed that using a standard weight of sample and, in particular, a standard particle size distribution for the sinter sample to be tested, significantly improved the sensitivity of the hardness measurement.

During furnace elimination studies, measurements of sinter hardness were made upon the sinter feed to the furnaces. During the test period, sinter hardness varied from 0.254 to 0.449 units and zinc elimination from the furnace charge increased 2.99 pct for each 0.1 unit decrease in sinter hardness within that range.

To study the relationships between sinter production variables and sinter hardness, seventeen independent variables were tabulated and used in a multiple regression analysis with sinter hardness as the dependent variable. In the computation, the independent variables were divided into three groups as follows: Group I—composition variables; i.e., zinc, iron, carbon, sulfur, etc. Group II— feed component variables; i.e, return fines, zinc oxide, calcine, etc. Group III—sinter burning variables; i.e., machine speed, wind box suction, etc.

The results of the analysis indicated that 62.7 pct of the total hardness variation could be accounted for by the changes in the composition variables, Group I.

It should be noted that, in Group II, only the calcine content of the feed and the residue fines content of the feed showed any significant relationship to hardness. Sinter hardness was also influenced to some extent by feed rate, air flow, and wind box suction.

Petrographic and X-ray diffraction techniques tentitively identified the major phases in the sinter as zinc oxide, zinc ferrite ($ZnO \cdot Fe_2O_3$), and zinc orthosilicate ($ZnO_2 \cdot SiO_2$). The analytical methods of Frenay, Collee, and Grodent detected and quantified these same phases. This technique was also used to determine the amounts of these same zinc-containing phases present at various stages of sinter reduction.

Based upon the observations made, the following conclusions may be drawn:

1) Use of a standard particle size distribution for the sinter sample subjected to the modified ASTM hardness test increases the sensivity of the test. Significant sinter hardness changes can be determined for laboratory and commercially produced sinters;

2) Of the variation in sinter hardness of commercially produced sinter, 62.7 pct is accounted for by changes in the chemical composition of the sinter. Increases in silica, iron, or lime content of the sinter increase sinter hardness. Increases in zinc or magnesia content in the sinter decrease sinter hardness;

3) Three major phases have been tentatively identified in the sinter structure: zinc oxide crystals, zinc ferrite grains, and a silicate network containing zinc oxide, alumina, lime, and magnesia. The reduction and elimination of zinc from the silicate network is more difficult to achieve than from the other two phases; and

4) Sinter chemical composition has a strong relationship both with sinter hardness and with zinc elimination from the electrothermic furnace. Sinter hardness is strongly correlated with furnace elimination. For the sinter hardness range from 0.254 to 0.449 units, each 0.10 unit decrease in sinter hardness increases furnace elimination of zinc 2.99 pct.

Relationship Between Sinter Hardness and Chemical Analysis of Sinters

Component	Comp. Range, pct	Increase in Hardness, per 1.0 pct addition	Probability of Effect, pct
Zn	51.5 to 59.8	−0.007	99
Fe	7.5 to 10.8	0.0191	99
SiO_2	6.5 to 10.0	0.0161	99
CaO	0.80 to 2.00	0.053	95
MgO	0.60 to 1.10	−0.114	95
Al_2O_3	—	not significant	—

And as the sun

This is only a sample of the many excellent technical papers presented at the Symposium. Your attention is drawn to the full proceedings which will be published shortly by Interscience. An announcement will appear in the JOURNAL OF METALS.

In addition to these and the other papers presented at the symposium, an international team consisting of 14 members from eight countries was organized to standardize the quality testing of agglomerates. The team is considering proposals of a drum apparatus which could be used to test any type of agglomerated product. All participants are to forward information concerning the methods they are now using with the hope that an agreement can be reached for an *international* drum test to standardize the testing of agglomerates. The team is expected to present a progress report at the 1962 meeting of the Blast Furnace, Coke Oven, and Raw Materials Committee.

And with the ending of the First International Symposium on Agglomeration, the almost 600 registrants pulled themselves out of their thoughts on pellets, nodules, briquets, sinter, and the like. It had been a very informative three days and left much to think about. Chairman W. B. Stephenson and all of the committees involved in planning the Symposium should be congratulated for a well-organized, efficiently-run, and very informative meeting. EJF

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SECRETARY—R. W. SHEARMAN
ASST. SECRETARY—D. C. JOHNSTON

NEWS OF YOUR PROFESSION

Metallurgical Society Adds Three Volumes To Conference Series

The Metallurgical Society has published four new technical volumes, which are issued through Interscience Publishers, Inc., of New York.

The new publications include volumes 7, 8, and 9 in the Society's Conference series, and a book on *Extractive Metallurgy of Copper, Nickel, and Cobalt*, which covers a symposium held in New York, February 15-18, 1960, and sponsored by the Society's Extractive Metallurgy Division.

Volumes 7 and 8, on *Physical Chemistry of Process Metallurgy*, contain the proceedings of a conference held in Pittsburgh, April 27-May 1, 1959. Volume 7 has 35 papers, Volume 8 contains 32 papers.

Volume 9 in the series contains the proceedings of a meeting sponsored by the Institute of Metals Division's Physical Metallurgy Committee at Estes Park, Colo., July 11-12, 1960. This book has two sections, one on *High Velocity Deformation*, and the other on *Shock Phenomena in Metals*.

Diffraction Conference Set For November In Pittsburgh

The annual Pittsburgh Diffraction Conference will be held November 1-3, at the Mellon Institute in Pittsburgh. Sessions will be devoted to metals and alloys, instrumentation, structures, polymers and fibers, refractories, and electron probe, including special sessions on electron diffraction and X-ray diffraction microscopy.

There will be an evening meeting, at which Dr. Peter J. W. Debye, emeritus professor of chemistry at Cornell University, will be the principal speaker. Further information may be obtained from the publicity chairman, T. B. Massalski, Mellon Institute, 4400 Fifth Ave., Pittsburgh 13, Pa.

METALLURGICAL SOCIETY'S FALL MEETING SCHEDULED OCT. 23-26 IN DETROIT, MICH.

Titanium in 1975 will be the title of one of the symposia at the Fall Meeting of The Metallurgical Society, which will be held at the Pick-Fort Shelby Hotel in Detroit, October 23-26.

The titanium symposium, which will be presented by the Institute of Metals Division, will include papers on *Background* by W. W. Minkler; *Production of Primary Titanium Products* by H. H. Kellogg; *Materials* by D. J. MacPherson; *Processes* by R. J. Rieppel; *Aerospace Applications* by Dana Morgan; and *Terrestrial Applications* by W. E. Lusby, Jr.

The Fall Meeting will be held in conjunction with the American Society for Metals' Congress and Metals Show which will be held in Detroit's Cobo Hall. The meeting will also coincide with the visit to the United States of the British Iron and Steel Institute, whose members will attend the Detroit meetings as invited guests of The Metallurgical Society and ASM. The British visit will last from October 17-November 7.

Each of The Metallurgical Society's three divisions will sponsor symposia at the meeting. The Institute of Metals Division, in addition to the symposium on titanium, will offer a symposium on *Refractory Metals*, consisting of four sessions; *Effects of Surface and Environment on Strength*, three sessions; *Theory of Work Hardening*, two sessions; and *High Purity Iron*, two sessions.

The Extractive Metallurgy Division will offer a symposium on *Nuclear Fuels Reprocessing*, which will consist of six papers on *Pyro Processing, Salts, Recycled Fuels, and Wastes;* and eight papers on *New Foreign and American Developments*. A second EMD symposium on *Vacuum Techniques in Extractive Metallurgy*, will consist of 14 papers.

Among the titles listed for presentation are *Arc vs. Electron Beam Melting in High-Vacuum Environments*, by R. P. Morgan and T. E. Butler; *Production Applications of the Electron Beam Heating Source to the Refractory Metals*, by E. F. Baroch and W. A. Aschoff; and *The Production of Pure Molybdenum and Its Alloys by Floating Zone Melting in Vacuum*, by J. A. Belk.

The Iron and Steel Division will offer a morning symposium on *Continuous Casting* consisting of at least three papers. The division will also present an afternoon symposium on *Mechanical Working of Cast Structure*, for which four papers are scheduled.

The complete program of the Fall Meeting will be published in the September issue of JOURNAL OF METALS.

Correction

In the July issue of JOURNAL OF METALS, the headline of a story on page 512 incorrectly stated that the Mechanical Working Conference would be held in Detroit. The conference will be held in Chicago, January 17, 1962 as was stated in the body of the story.

J. T. Norton Awarded 1961 Plansee Plaque

John T. Norton, dean of the graduate school and professor of physics at Massachusetts Institute of Technology, was awarded the 1961 Plansee Plaque at the opening session of the Fourth International Plansee Seminar, which was held June 20-24 in Reutte, Tyrol, Austria. The topic of the seminar was *Powder Metallurgy in the Nuclear Age*.

A member of AIME, Dr. Norton received the plaque for "outstanding contributions to the art and science of powder metallurgy." He is the first American to receive the award.

Dr. Norton is recognized for his fundamental work on the structure of metals and the relationship between structure and properties. He initiated and stimulated basic research on the structure and properties of metallic carbides, borides, and related compounds. A summary of the Plansee Seminar will appear in a forthcoming issue of JOURNAL OF METALS.

New England Regional Conference Topic Is Basic Metals Industry

The Basic Metals Industry—Technical and Economic Prospectives was the theme for the 15th New England Regional Conference held at the General Electric Co.'s Research Laboratory, Schenectady, N. Y., May 18-19. The nine-paper program emphasized the past, present, and future of the aluminum, copper, and steel industries. In addition to the excellent papers presented at the conference, two panel discussions provided for audience participation.

For a background, a history of the basic metal industry was presented by the speakers during the opening session. In his paper on *Aluminum*, L. P. Favorite, Aluminum Company of America, traced the light metal industry's development from the time C. M. Hall started experimenting in his woodshed at Oberlien, Ohio to the present. O. C. Herfindahl, Resources for the Future, Inc., discussed the *Copper* industry. The author stated that competition from other materials has hindered somewhat the growth of the industry, but basically the industry is stable from a world market view point. The *Steel* industry was the subject of R. D. Woodward, Bethlehem Steel Co. It was pointed out that the growth of the steel industry was approximately the same as that of the Gross National Product (GNP) from 1900-56, and the current *plateau* in the growth curve is temporary.

Speakers at the afternoon session concerned themselves with the question—*What is the basic metals industry?* W. V. Lidgerwood, Aluminium Limited Sales, Inc., discussing *Aluminum* stated that overproduction in the industry is causing fierce competition. Apparently the answer lies in the vast amount of research and development being carried out by the industry. *Copper* was discussed by J. S. Smart, Jr., American Smelting and Refining Co., and President of The Metallurgical Society. The author emphasized the worldwide character of the industry, and compared the amounts of ore necessary to produce one ton of copper to the amounts necessary for producing aluminium or steel. Rev. W. T. Hogan, Fordham University, discussed the *Steel* industry. The author pointed out that it is difficult to assess the capacity of the industry because of the rapid technological changes taking place, but stated modernization is imperative.

The panel discussion which followed included speakers from the morning and afternoon sessions, plus E. Barnes, Scovill Manufacturing Co.; Mr. Hildy, Electric Boat div.,

(Continued on page 578)

Theodore B. Counselman

Theodore B. Counselman Receives Alumni Medal From Columbia Univ.

Theodore B. Counselman, consulting metallurgical engineer and secretary-treasurer of Behre, Dolbear, and Co., has been awarded Columbia University's Alumni Medal for "service of exceptional merit" to the University and to the School of Engineering of which Mr. Counselman is a graduate in the class of 1910. The medal was presented on Commencement Day, June 6.

A member of AIME for 47 years, Mr. Counselman has served as chairman of the Chicago and New York sections; Chairman of the National Membership Committee; Chairman of the Blast Furnace and Raw Materials Committee; Chairman of the Iron and Steel Division. He has also been a Director and member of the Executive Committee, as well as Vice President of AIME.

Mr. Counselman is currently a director of Engineers Joint Council, and Chairman of the Admissions Committee.

He will retire this year from the presidency of the Columbia Engineering School Alumni Association which he has served in various capacities for a number of years.

Mr. Counselman began his engineering career with sulfur exploration in Texas, continued with the Consolidated Copper Co., in Arizona, and he was concerned with the magnetic taconite development of the Mesabi Iron Co. in Minnesota. For 28 years, he was a member of the Dorr Co., concerned with iron ore beneficiation, cement manufacture, chemical processes, and fluo-solids roasting equipment.

Steel Symposium Set In Czechoslovakia September 10-18

An international symposium on corrosion resisting steels and alloys will be held in Czechoslovakia, September 10-18, 1961.

Delegates to the convention will arrive in Praha on September 10. The next three days (September 11-13) will be devoted to lectures and discussions of foreign and Czechoslovak delegates. The program includes *Physical Metallurgy of Stainless Steels and Alloys; New Developments in Ferritic Stainless Steels; New Developments in Austenitic Stainless Steels; High Quality Stainless Steels;* and *Metallurgical and Technological Aspects of Stainless Steels and Alloys.*

On September 14, the delegates will travel by plane to Brno (Brunn). The group will visit the International Fair at Brno, and tour a factory producing machinery for the chemical industry. This visit will last until September 16. On September 18, the group will visit a large metallurgical plant in the Ostrava region. They will leave by plane the same day for Praha, where they will visit the Research Institute of Material on September 19.

Foreign delegates will have the opportunity to visit historical places and cultural institutions in the capitol of Czechoslovakia, and to take part in an excursion to the mountains.

Persons interested in attending the symposium may contact Dr. Ing. Jaroslav Pluhar, chairman of the Preparatory Committee of the Symposium; The Czechoslovak Academy of Sciences, Praha 1, Czechoslovakia.

Italian Metallurgists Will Meet in Milan October 7-9, 1961

The presentation of the Fourth Guido Donegani Gold Medal Award will be one of the features of the fifth meeting of the Centro Leghe Leggere of the Associazione Italiana di Metallurgia which will be held in Milan, Italy, October 7-9. The main topic of the meeting will be *Surface Treatments of Aluminum.*

During the meeting particular attention will be given to the anodic oxidation and to the chemical and electrochemical preparation problems.

It is expected that during the meeting, the events will be simultaneously translated into English, French, and German.

Persons wishing to attend the meeting should send registrations to the Secretary, Associazione Italiana di Metallurgia, Via della Moscova 16, Milan, Italy.

Pacific N.W. Minerals Conference Held in Spokane

Sunny Spokane and the comfortable Davenport Hotel were hosts this spring to the 1961 Pacific Northwest Metals and Minerals Conference. Co-sponsors of this year's gathering, held April 13-15, were AIME's Columbia section and the Inland Empire chapter of the American Society for Metals. The comprehensive nature of the program, which ranged from geophysics through metalworking to economics, attracted over two hundred registrants who represented a cross-section of mineral and metals industry and education in the northwest.

Primary Metalworking

In addition to sessions on mining, geology, and industrial minerals, the opening day of the Conference included two sessions on primary metalworking. Leading off in the morning was a description of copper refining facilities at Anaconda's Great Falls reduction plant, authored by R. J. Lapee, The Anaconda Co., N. Y. Next, D. R. Bristol, Daystrom Corp., La Jolla, Calif., summarized the status of efforts to apply automation and modern techniques in instrumentation to the steel industry with emphasis on rolling mill operations. He also described various proposals for more advanced installations in the near future.

Many of the registrants were to be found at the ASM luncheon on Thursday, where introductory remarks were ably delivered by Dr. C. E. Swartz, a consulting metallurgist and vice president of ASM. Noting that the conference was a cooperative effort of AIME and ASM, Dr. Swartz reminded registrants that AIME, as a professional society, and ASM, as an association of the metals industry in the US, also complement each other through providing services uniquely suited to their respective members. The featured speaker at the luncheon was J. A. Laberee, E. I. du Pont de Nemours & Co., Palo Alto, Calif., who discussed *The Disappearance of the Obvious*. His concern is primarily with the tendency in American society to obscure the relationship between cause and effect, particularly in the case of individual actions, and the de-emphasis of the importance of basic principles in human relations. As symptoms of this trend, Mr. Laberee cited the decline of personal responsibility, the decreasing importance of individual effort, the confusion of equality of ability with equality of opportunity, and a preoccupation with the Communist threat to American society.

Attendants at the afternoon session on metalworking heard a thorough and most interesting report on fabrication of columbium alloys and other refractory metals by E. F. Baroch, Wah Chang Corp., Albany, Ore., and an analysis of forging quality by R. W. Hargis, Isaacson Iron Works, Seattle, Wash.

A well-attended social hour and an informal buffet preceded the Thursday evening panel discussion of *Gold in the Monetary System*. In addition to moderator Hollis Dole, Oregon State Dept. of Geology and Mineral Industries, Portland, the panel included V. C. Wansbrough, Canadian Metal Mining Assn., and Dr. Eugene Clark, Dean, School of Economics and Business, Washington State Univ.

Among the subjects of Friday's technical sessions were utilization of concrete in mining operations, geology, and secondary metalworking. The latter included, in the morning, a report on swage forming of metals for nuclear applications, by F. B. Quinlan, General Electric Co., Richland, Wash., discussion of hot extrusion techniques for fabricating fuel elements by J. W. Nickolaus and C. H. Shaw, General Electric Co., Richland, and a report on applications for recent developments and new techniques in metal forming, by Frank Sutch and E. L. Stone, Boeing Airplane Co., Seattle.

AIME Luncheon

At the AIME luncheon on Friday, registrants found that areas of common interest still exist in spite of their diverse background. This was amply demonstrated when L. E. Elkins, production research director, Pan-American Oil Co., Tulsa, Okla., and President-Elect of AIME, told how *AIME Members Provide the Keystone to Fuel Security*. After summarizing trends in the discovery and exploitation of oil reserves in the US, Mr. Elkins pointed out that fuel security in future years will be increasingly dependent on development of techniques for improving secondary recovery of existing, as well as, newly-discovered deposits. (Secondary recovery refers to that portion of a deposit which is not retrievable simply by pumping from the reservoir.) He then described the various methods which are presently employed in secondary recovery, noting that the wide variation in characteristics from one basin to another requires extensive innovation and the application of many branches of science and engineering to the solution of recovery problems.

Presented at the final metalworking session were the following papers: *Electron Beam Zone Refining of Tungsten*, Lloyd Bazant, U.S. Bureau of Mines, Albany, Ore., *Fabricating Structural Parts from Molybdenum-½ pct Titanium Sheet*, by D. A. Honebrink, Boeing Airplane Co., Seattle, and *High Energy Impact Forming of UO_2*, by D. W. Brite, R. J. Anicetti, and R. G. Wheeler, General Electric Co., Richland.

Flagging spirits were revived during both days of technical sessions by the coffee breaks provided through the efforts of AIME Women's Auxiliary members, who also organized a cocktail hour and luncheon for the ladies in attendance at the Conference.

Plant Visits

The final morning of the meeting was devoted to plant visits with registrants being offered a tour of the Lucky Friday Mine and Mill in Mullan, Idaho, a visit to metallurgical operations at the Bunker Hill Co., in Kellogg, Idaho, and an inspection of USAF Air Defense Command missile site construction on the outskirts of Spokane, Wash.

Space permits mention here of only a few of the many AIME and ASM members who contributed to the success of the 1961 Pacific Northwest Metals and Minerals Conference: A. Y. Bethune, chairman, Columbia section, AIME; F. W. DeMoney, chairman, Inland Empire chapter, ASM; W. D. Nesbeitt and F. W. DeMoney, conference co-chairmen; J. J. Quinlan, program chairman, F. N. Marr, arrangements chairman; and A. E. Weissenborn, publicity chairman.

Dr. Benjamin Lustman Nominated by AIME For Niels Bohr Gold Medal

Dr. Benjamin Lustman has been nominated by AIME to receive the Niels Bohr International Gold Medal which will be presented in October by the Institution of Danish Civil Engineers. The award is given every three years to an engineer or physicist in honor of his outstanding work for the peaceful utilization of atomic energy.

Dr. Lustman received his B.S., M.S., and D.S.C. in metallurgical engineering from Carnegie Institute of Technology. He has been engaged in research and development of nuclear reactor materials since 1949 for Westinghouse Electric Corp., and is currently manager, reactor metallurgy, of the Bettis Atomic Power Laboratory. He has been especially active in the development of zirconium base alloys for cladding materials on fuel elements and the development of uranium dioxide as a highly radiation resistant fuel material.

AROUND THE SECTIONS

Connecticut section, AIME, met June 7, to hear an address by Clarence Bieber, head of the special alloy section, Bayonne Laboratary of International Nickel Co. Mr. Bieber spoke on *Creative Metallurgy*. He described the development of superalloys for turbine blades of jet aircraft, pointing out the prime requisites of strength, ductility, resistance to corrosion, impact strength, structural stability, ease of manufacture, low cost, service performance, weldability, low density, coefficient of expansion, thermal conductivity, modulus of elasticity, and machinability.

Elected as officers for the 1961-62 term are: *Chairman*: William R. Johnson, Associated Spring Co., *Vice-Chairman*: Harold M. Cobb, Olin Mathieson Chemical Corp., *Secretary*: Malcolm F. Burr, Chase Brass & Copper Co., *Treasurer*: J. Bruce Wagner, Yale University, and *Member-at-Large*: Fred A. Besel, Anaconda American Brass Co.

Elmer S. Barnes, Scovill Mfg. Co. will serve as chairman of publicity, and Herbert A. Ball, Olin Mathieson Chemical Corp., will serve as chairman of membership.

San Francisco section met June 14 at the Engineer's Club with 86 members and guests attending. Guest speaker for the evening was Glenn H. Hanson of Allis Chalmers Manufacturing Co., Milwaukee. He discussed the *Grate-Kiln System and Its Application in Iron Ore Agglomeration*.

The section's next meeting will be held on October 11.

Southeast section, AIME, under the leadership of J. W. Nicol, section chairman, is making plans to establish a metallurgical division to function for the present within the ranks of the Southeast section.

This is a cooperative venture and has the full support of the local leaders in both the mining and metallurgical field.

As a kickoff meeting, the September meeting will feature a metallurgical paper. The October meeting is the AIME-ASME Joint Solid Fuels Conference. In November, a paper on Coke will be presented. The predominantly metallurgical slant of the fall meetings is to stimulate interest within the metallurgical group. This will be the first such section organized. In the Birmingham area, interdependent activities in mining and metallurgy are closely integrated.

Among those attending the first planning session of this combined mining and metallurgical section were: J. W. Nicol, U.S. Pipe and Foundry Co.; E. P. Reed, John Hunt, L. Sam Chabot, Jr., and Walter Kirkwood, all of Tennessee Coal and Iron div., U.S. Steel Corp.; Walter Nielsen and M. M. Marchich, U.S. Pipe and Foundry Co.; and James E. Brown, Jr., Southern Generating Co.

Ohio Valley section will hold its *National Officers Night* on October 12. Carleton C. Long, director of research, St. Joseph Lead Co., has tentatively accepted an invitation to be the guest speaker. He is expected to speak on *Cooperative Research by Metal Trade Associations*.

BOOKS

(Continued from page 522)

Trends and Problems of the European Steel Industry (See JOURNAL OF METALS, May 1961, p. 342), made under the auspices of the ECE Steel Committee.

Inquiries should be directed to Publishing Service, United Nations, New York, N. Y.

Professional Manpower and Education in Communist China by Leo A. Orleans, National Science Foundation, 260 pp., $2.00, 1961—This report sees Communist China's scientific effort geared to meet immediate technological demands, its education undergoing rapid change and expansion, and its development as a major industrial power handicapped by its population problem.

Copies of this report are available from the *Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C.*

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I am interested in joining the Society. Please send further information and an application for membership.

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N. E. Regional

(Continued from page 576)

General Dynamics Corp.; J. Douglas, Republic Foil Co.; and J. J. Lennon, Amco div., American Metal Climax. Moderator R. P. Carreker, General Electric Co., initiated the lively discussion which followed.

Informal was the word for the cocktail hour and the dinner held at the Hotel Van Curler that evening. A buffet-style dinner helped to attain an air of informality which lasted throughout the evening. Guest speaker J. C. Fisher, General Electric Co., talked about *Growth of Science and Technology in Underdeveloped Countries*. The talk was most stimulating and brought forth many questions from the floor during the discussion which followed.

The next morning it was back to business and the subject was the future of the basic metals industry. Forecasts for the *Aluminum* industry were made by L. W. Eastwood, Metals div., Olin Mathieson Chemical Corp. The speaker predicted that domestic aluminum consumption will increase, exports will decrease, and imports are expected to remain at least the same. *Copper's* future was discussed by C. H. Moore, Copper Products Development Association. The author told of the integrated effort in research and development of the copper industry, and explained the purpose of the Copper Products Development Association (CPDA). More details on this organization can be found in the February 1961 issue of JOURNAL OF METALS, p. 115. T. T. Magel, Allegheny Ludlum Steel Corp., discussed the future of the *Steel* industry. He indicated that the increasing amount of basic research being carried out, and the arrival of new processes appear to indicate that the future of the steel industry looks bright.

R. P. Carreker again moderated the panel discussion which included speakers from the morning session plus E. J. Lally, American Metal Market; I. Lipkowitz, Reynolds Metals Co.; and T. Velfort, Copper and Brass Research Association. At the conclusion of the discussion, the registrants left the research laboratory for a tour of Watervliet Arsenal, Watervliet, N. Y.

Conference Chairman C. M. Adams, Jr. and the committee should be congratulated on presenting such a timely and interesting program.

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MEMBERSHIP

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Metallurgical Society of AIME

Total AIME members on June 30, 1961, was 35,086; in addition 2,095 Student Members were enrolled.

ADMISSIONS COMMITTEE

W. L. Brytczuk, Chairman; S. C. Carapella, Jr.; J. W. Hanley; T. D. Jones; H. C. Larson; Harold Margolin; Shadburn Marshall.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

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Goldstein, Ernst M., Newfield, N. J.
Pozefsky, Leo, Bethlehem, Pa.

Southwest Mineral Industry Conference Held in Las Vegas

AIME members found a *sure thing* in Las Vegas if they attended the 1961 Southwest Mineral Industry Conference, held April 24-25 at the Stardust Hotel. Sponsored by the Nevada section, the Conference also featured participation by Metals and Mining Branches of the Southern California section, the San Francisco section, and the Industrial Minerals Division, Society of Mining Engineers, all of AIME. The program was distinguished by the wide variety of subjects under discussion, including industrial minerals, extractive metallurgy, applications of nuclear energy, geology, and physical metallurgy.

At the opening session for all participants on Monday morning, general chairman Victor Howard, Basic Incorporated, welcomed registrants before introducing the chairmen of various subcommittees. The opening talk was presented by Dr. Lewis Fussel, Jr., Edgerton, Germeshausen, & Grier, Inc., who discussed *The Future of Science and Technology*. Noting that it is customary for speakers on this subject to list some predictions for the future, Dr. Fussell quickly declared his conviction that man would find economical means for converting salt water to fresh, would recover minerals, food, and limitless energy from the seas, would conquer such diseases as cancer, and would travel to the moon and beyond and return. He just as quickly confessed his inability to predict when these things will occur, however. This uncertainty was attributed to our being in the midst of a full-fledged technological revolution, with one important result being the reduction and probable elimination of the lead time between scientific discoveries and their application. Dr. Fussell cited as most urgent, under the circumstances, the need for a better means of communication.

Members and guests attending the conference were greeted at a welcoming luncheon on Monday by the Hon. Grant Sawyer, Governor of Nevada, who reiterated the large part played by the mineral industries in populating the state as well as their continued importance to Nevada and all of the southwest.

An interesting paper was delivered at the extractive metallurgy session on Monday afternoon by Prof. W. H. Voskuil, University of Nevada. Titled *Steel Manufacture in the Southwest: Market and Raw Material Potentials*, Prof. Voskuil's analysis showed that, taken as an economic whole, the southwest is a net importer of basic metal for fabrication. He suggested, however, that the only means for increasing raw metal production lay in such developments as cheaper fuels, new sources of raw materials, and the feasibility of new techniques such as the H-iron and other direct reduction processes. He also commended to the consideration of metal producers the possibility of using cupolas for iron production.

Conferees were addressed at luncheon on Tuesday by R. R. McNaughton, 1961 President of AIME, who attended the Conference.

The first paper presented at the Tuesday afternoon physical metallurgy session was a review of properties of electron beam melted metals and alloys for nuclear applications by C. D. Hunt, Temescal Metallurgical Corp., Berkeley, Calif. Discussing commercial applications of titanium metals, L. J. Barron, E. I. duPont de Nemours & Co., listed resistance to contamination and abrasion-erosion, lower prices, and improved fabricating techniques, in addition to corrosion resistance, as contributing factors in commercial growth of these materials. He predicted that titanium would be a major non-ferrous material of construction by the end of the sixties. Centrifugal casting of vacuum melted tungsten alloys in graphite molds rotating about a vertical axis was described by R. G. Hardy, J. L. Gunter, and T. A. Hamm, Oregon Metallurgical Corp., Albany. The final paper in this session was a report on improved austenitic manganese steels for abrasion resistant applications, by T. E. Norman, Climax Molybdenum Co., Denver, Colo. He described two new types of steel, one of which exhibits improved abrasion resistance while retaining the high ductility typical of Hadfield steel. The second type, which has a lower alloy content, combines moderate ductility with markedly improved abrasion resistance. Fields of application for both of the new materials were listed.

Among the many persons to whom the more than 300 registrants were indebted for a successful conference were the following: Victor Howard, general chairman; E. Don Dilling, general vice-chairman; W. G. Flangas, program chairman; F. E. Love, arrangements chairman; and L. J. Hartzell, publicity chairman.

NECROLOGY

Date Elected	Name	Date of Death
1944	Allsbrook, S. T.	May 21, 1961
1952	Burgess, Thomas M.	June 2, 1961
1947	Crumbaugh, R. C.	Unknown
1910	Daniell, John, Jr. (Legion of Honor)	July 7, 1960
1916	Eichrodt, Charles W.	May 31, 1961
1914	Fitch, Cecil	Apr. 16, 1961
1920	Hunter, Matthew A.	Unknown
1952	Knill, Raymond R.	Mar. 25, 1961
1957	Markle, Frank B.	Apr. 17, 1961
1920	Morse, Roy Robt.	Unknown
1917	Naramore, Chester	Jan. 30, 1961
1944	Nock, Chas. J.	June 6, 1961
1953	Otis, Russell M.	Unknown
1958	Peller, Leonard	Unknown
1918	Thum, E. E.	Apr. 10, 1961
1913	Tremoureux, Roy E.	May 11, 1961
1955	Wallace, Demi F.	Unknown

Coming Events

Aug. 23-25, 1961, AIEE Pacific General Meeting, Hotel Utah, Salt Lake City, Utah.

Aug. 30-Sept. 1, 1961, AIME Third Technical Conference on Semiconductors, Ambassador Hotel, Los Angeles.

Aug. 31, 1961, Chicago Section, NOHC, Annual Golf Outing, Gleneagles Country Club.

Sept. 14-15, 1961, Joint Engineering Management Conference, Roosevelt Hotel, New York.

Sept. 17-20, 1961, Commemoration of the 50th Anniversary of Froth Flotation, Cosmopolitan Hotel and Brown Palace Hotel, Denver, Colo.

Sept. 22, 1961, Cleveland Section, NOHC, All-Day Technical Meeting, Sheraton-Cleveland Hotel, Cleveland, Ohio.

Oct. 2, 1961, Chicago Section, NOHC, Fall Technical Dinner Meeting, Phil Smidt's Restaurant, Hammond, Ind.

Oct. 2-3, 1961, 29th Annual Meeting, Engineers' Council for Professional Development, Sheraton Hotel, Louisville, Ky.

Oct. 5-6, 1961, Southwestern Section, NOHC, Annual Fall Meeting, President Hotel, Kansas City, Mo.

Oct. 17-Nov. 7, 1961, Visit of the Iron & Steel Institute (United Kingdom) to the United States.

Oct. 20, 1961, Eastern Section, NOHC, 15th Annual Technical Conference, Warwick Hotel, Philadelphia.

Oct. 23-26, 1961, Fall Meeting of The Metallurgical Society of AIME, Pick-Fort Shelby Hotel, Detroit.

Nov. 1-3, 1961, Annual Pittsburgh Diffraction Conference, Mellon Institute, Pittsburgh, Pa.

Nov. 3, 1961, Pittsburgh Section, 16th Off-the-Record Meeting, Penn-Sheraton Hotel, Pittsburgh, Pa.

Nov. 13-16, 1961, Seventh Annual Conference on Magnetism and Magnetic Materials, Hotel Westward Ho, Phoenix, Ariz.

Nov. 14, 1961, Buffalo Section, NOHC, 12th Annual Meeting, Royal Connaught Hotel, Hamilton, Ont., Canada.

Nov. 29-30, 1961, Iron and Steel Institute (U.K.) Fall General Meeting on Future of Ironmaking in the Blast Furnace, London.

Dec. 4, 1961, Annual Meeting, Arizona Section, AIME, Pioneer Hotel, Tucson, Ariz.

Dec. 6-8, 1961, 19th Electric Furnace Conference, Penn-Sheraton Hotel, Pittsburgh, Pa.

Jan. 17, 1962, AIME Mechanical Working Conference, Pick-Congress Hotel, Chicago, Ill.

Feb. 18-22, 1962, AIME Annual Meeting, New York, N. Y.

Apr. 9-11, 1962, NOHC Blast Furnace, Coke Oven, and Raw Materials Conference, Sheraton-Cadillac Hotel, Detroit, Mich.

Apr. 12-14, 1962, AIME Pacific Southwest Minerals Industry Conference, Palace Hotel, San Francisco, Calif.

April 26-28, 1962, AIME Pacific Northwest Metals and Minerals Conference, Ben Franklin Hotel, Seattle, Wash.

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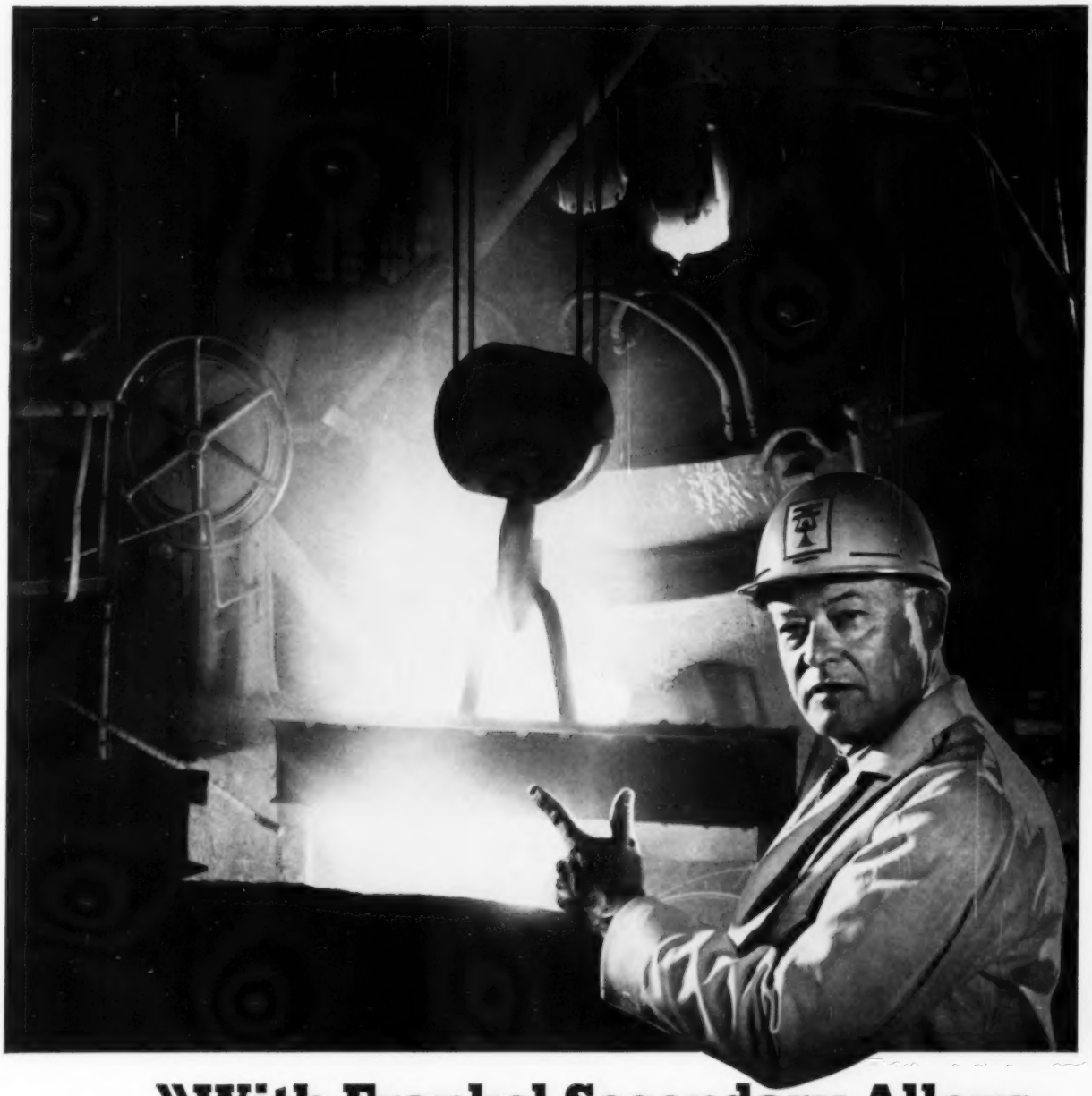

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